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Color Removal from Kraft Mill Effluents by Ultrafiltration

Champion International Corp.
Hamilton, OH

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Color removal from kraft mill effluents by ultrafiltration (UF) has been examined during this program. A 3-stage, nominal 37.9 m^3 (10,000 gpd) UF pilot plant was operated on caustic extraction filtrate for several months. Extensive evaluation of spiral-wound UF modules was carried out prior to staged system operation in single module tests. During these tests feed pretreatment and prefiltration options were investigated and the effects of a range of operating parameters on module flux performance were studied. A second module configuration, tubular assemblies, was also tested. All field tests were performed at the Canton, North Carolina Mill of Champion International.

Non-cellulosic UF membranes were evaluated in laboratory tests before field trials were initiated. The preferred membrane was cast from a polysulfone formulation.

Spiral modules were severely fouled by species present in white water recycle. Tubular modules, however, exhibited stable, economically-viable flux performance.

Color removal by the tubular UF membranes ranged from 97% to 99% when calculated on a concentrate basis. Projections based on process data indicate UF results in an overall color reduction of 91% (mass basis) for caustic extraction filtrate.

Conceptual designs and economic analyses were developed for treatment systems with capacities of 3,790 m^3/day (1 MM gpd) and 7,980 m^3/day (2 MM gpd). Additionally, caustic extraction filtrate and decker effluent stream characteristics were monitored and qualitative assessments of ultrafiltrate and UF concentrate recycle within a kraft mill were made.

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February 1980

COLOR REMOVAL FROM KRAFT MILL EFFLUENTS BY ULTRAFILTRATION

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FOREWORD

When energy and material resources are extracted, processed, converted, and used, the related pollutional impacts on our environment and even on our health often require that new and increasingly more efficient pollution control methods be used. The Industrial Environmental Research Laboratory - Cincinnati (IERL-Ci) assists in developing and demonstrating new and improved methodologies that will meet these needs both efficiently and economically.

A field demonstration of color removal from kraft mill effluent streams by ultrafiltration is discussed in this report. Technical and economic assessments of caustic extraction filtrate, pine decker and hardwood decker effluent treatment by both spiral-wound and tubular ultrafiltration modules are presented. It is hoped that the results of this study will lead to continued research in the field of color removal by membrane processes and eventually provide the pulping industry with a cost-effective method for resource recovery and pollution abatement with these complex effluent streams.

The Food and Wood Products Branch of the Industrial Environmental Research Laboratory should be contacted for further information on this subject.

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ABSTRACT

Color removal from kraft mill effluents by ultrafiltration (UF) has been successfully demonstrated during this program. A 3-stage, nominal 37.9 m^3/day (10,000 gpd) UF pilot plant was operated on caustic extraction filtrate for several months. Extensive evaluation of spiral-wound UF modules was carried out prior to staged-system operation in single module tests. During these tests feed pretreatment and prefiltration options were investigated and the effects of a range of operating parameters on module flux performance were studied. A second module configuration, tubular assemblies, was tested in both 12.7 mm (0.5 in) and 25.4 mm (1 in) diameter tubes. All field tests were performed at the Canton, North Carolina Mill of Champion International.

Non-cellulosic ultrafiltration membranes were evaluated in laboratory tests before field trials were initiated. The preferred membrane was cast from a polysulfone formulation.

Spiral-wound modules showed severe flux loss within a few hours exposure to the waste stream. Membrane surface analysis identified the main stream foulants as kaolinite clay, starch and titanium dioxide. These foulants were the result of white water recycle from the paper mill back to the pulp mill. Pretreatment and prefiltration techniques were ineffective in preventing "slime" layer formation by these species.

Tubular modules exhibited high, stable process flux and recoverable water flux characteristics. Membrane surface fouling was not observed with tubular modules. These modules operate under more turbulent flow than spiral-wound modules reducing "slime" layer formation. Average flux values when processing caustic extraction filtrate were 2.87 m^3/m^2-day (70 gfd) at a 1.2X concentration, 2.26 m^3/m^2-day (55 gfd) at a 10X concentration and 1.03 m^3/m^2-day (25 gfd) at a 50X concentration. These data were recorded at 50°C, 5.1 atm (75 psig) inlet pressure and a circulation flowrate through 25.4 mm diameter tubular assemblies of 136 m^3/day (25 gpm).

Color removal by the non-cellulosic ultrafiltration membranes ranged from 97% to 99% when calculated on a concentrate basis. Projections based on caustic extraction filtrate process data indicate ultrafiltration would result in an overall color reduction of 91% (mass basis) for this stream.

Conceptual designs and economic analyses were developed for treatment systems with capacities of 3,790 m^3/day (1 MM gpd) and 7,980 m^3/day (2 MM gpd). Both tubular and spiral-wound module systems were analyzed with process streams of caustic extraction filtrate and, pine and hardwood decker effluent. For tubular systems installed capital costs ranged to $4 MM. Operating costs ranged to $2.52/metric ton of pulp produced per day (to $2.27/ton). Spiral-wound module system costs were based on idealized systems and are not achievable given today's technology. Capital costs for spiral-wound module systems were projected to be ~50% of tubular system costs. Treatment costs were estimated to range to $1.89/metric ton of pulp (to $1.72/ton).

Additionally during this program, caustic extraction filtrate and decker effluent stream characteristics were monitored and qualitative assessments of ultrafiltrate and UF concentrate recycle within a kraft mill were made.

This report was submitted in fulfillment of Grant No. S804312-011 by Champion International and the Walden Division of Abcor, Inc. under the sponsorship of the U.S. Environmental Protection Agency. This report covers a period from March 29, 1976 to August 25, 1978, and work was completed as of September 29, 1978.

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ENGLISH-METRIC CONVERSION TABLE

To convert from	To	Multiply by
Atmosphere	Pound per sq. inch	14.7
Cubic meter	Cubic feet	35.31
Cubic meter	Gallon	264.2
Cubic meter per day	Gallon per day	264.2
Cubic meter per day	Gallon per minute	0.183
Cubic meter per sq. meter-day	Gallon per minute per sq. ft.	0.17
Cubic meter per sq. meter-day	Gallon per sq. ft-day	24.39
Kilogram	Pound	2.205
Kilo Watt	Horsepower	1.341
Meter	Feet	3.281
Meter	Inch	39.37
Square meter	Square feet	10.76
Square meter	Square inch	1,550

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SECTION 1

INTRODUCTION

BACKGROUND

The 119 kraft pulp mills (1) in the United States produce about 85% of the chemical wood pulp consumed. In an integrated pulping and paper making operation, a substantial volume of waste water is discharged, typically about 125 m^3/metric ton of pulp (30,000 gal per ton of pulp). Of concern are the pH, temperature, suspended solids, BOD and color loading of this effluent. Conventional and generally available techniques are adequate in most cases for waste treatment except for color removal. It may also be noted that conventional waste treatment does not provide for water reuse, and as such, is not conducive to eventual closed-loop operations.

Color bodies found in pulp and paper mill wastes are resistant to biological degradation. Consequently, new treatment techniques for color removal are undergoing active development and actual plant scale demonstration. Processes developed include chemical precipitation (2-11), including lime precipitation and alum precipitation, adsorption (12-15), oxidation (16-19) and reverse osmosis and ultrafiltration (20-30). Rapid infiltration (31) involving percolation of the effluent through the ground has been demonstrated.

The reduction of effluent color by modification of pulping and bleaching sequences has been the subject of extensive development. Segregation of mill waste streams is often practiced and it is likely that segregation of wastes by color content will eventually be required for adequate waste treatment. For example, in Champion Papers North Carolina Mill, about 60% of the total mill color effluent is present in about 3,790 to 7,580 m^3/day (1 to 2 MM gpd) of the pine bleachery first-stage caustic extraction filtrate. This flow amounts to about 2% to 5% of the total mill effluent, yet removal of color from this stream could reduce total effluent color by 60%.

The second most important controllable source of color in a kraft mill is the pulp washing decker effluent. This waste stream is present in all pulp mills, while the pine caustic extraction filtrate is found only in mills producing bleached pulp. At the North Carolina mill, approximately 7,580 m^3/day (2 MM gpd) of mixed pine and hardwood decker effluents are currently discharged. This waste contributes about 20% of the total mill effluent color.

In an EPA-supported program (Project No. S800261) (32) Champion International demonstrated an ultrafiltration process for kraft mill effluent color removal on selected pulping and pulp bleachery process effluents. The treatment of pine caustic extraction filtrate of a pulp bleachery and the decker effluents from unbleached pulp washing were examined in pilot scale studies. These two streams contribute about 80% to 90% of the color discharged from an integrated kraft mill. The results of this study are detailed in Champion's final report to EPA, dated May 1973 (32).

The major problem encountered in the original program involved membrane cartridge reliability and life. The spiral wound cellulose-acetate membranes employed were susceptible to cartridge plugging by suspended solids and membrane surface fouling by fine colloidal matter. The plugging problem was solved by use of cartridges with special flowchannel spacers. Trouble-free operation was achieved with minimal feed prefiltration, at least from the point of view of cartridge plugging.

More troublesome, however, was membrane surface fouling, which resulted in low membrane flux (unit capacity) and necessitated frequent membrane cleaning. Especially important in promoting membrane fouling were the feed pretreatment steps required to protect the cellulose-acetate membranes from chemical degradation. This pretreatment included temperature reduction (from about 60°C to 38°C) and pH reduction (from about pH 11 to pH 7). These two steps significantly reduced the stability of the colloidal material in the influent, resulting in rapid membrane fouling.

It was concluded that a membrane which could operate at the raw influent pH and temperature would offer significant process improvements:

1) reduced membrane fouling, hence higher flux and less frequent membrane cleaning; and

2) elimination of feed pretreatment, i.e. cooling and neutralization.

These improvements would lead to major benefits in terms of both increasing process reliability and decreasing process capital and operating costs.

After the original EPA-sponsored program, Champion conducted two additional related studies. The first demonstrated the technical effectiveness of spiral wound membrane modules with improved flow-channel spacers. These membrane modules exhibited the ability to process the effluents of concern without module plugging, and the accompanying mechanical failure, observed in the prior EPA-supported program.

The second program was sponsored to evaluate new non-cellulosic membranes developed by the Walden Division of Abcor, Inc. In this program, it was clearly demonstrated that membrane materials suitable for treatment of the raw effluents without pretreatment were becoming available. The results on membrane flux, color body removal effectiveness and cleaning ability were clearly superior to those obtained with the cellulose-acetate membranes employed earlier. In a parallel membrane development program at Columbia University, another membrane suitable for treatment of pulp mill effluents had been developed.

Based on the prior EPA program results, the module mechanical studies and the availability of these new membranes, Champion applied for a demonstration grant renewal, the results of which are reported here.

The program described in this report encompasses a two-year study for the development and demonstration of ultrafiltration as a process for color removal from kraft mill effluents. The site of the project was Champion Papers' Canton, North Carolina Mill, which is a typical integrated kraft mill.

The program included four major elements:

1. Evaluation and selection of non-cellulosic membrane materials and module geometry.
2. Selection and evaluation of feed pretreatment alternatives.
3. Modification of the 37.9 m^3/day (10,000 gpd) ultrafiltration system originally built for EPA project no. S800261.
4. Evaluation of non-cellulosic membranes in the modified 37.9 m^3/day (10,000 gpd) pilot system.

Additional studies were undertaken to develop: estimated operation and capital costs as well as space and energy requirements for several flow size units; bases for disposal of the concentrated wastes; and, the potential for reuse of the permeate water.

Briefly, the project objectives have been threefold.

1. To demonstrate with commercially available equipment the effectiveness of ultrafiltration to reduce color in the first-state pine bleachery caustic extraction filtrate and pulp washing decker effluents to low levels.
2. To demonstrate the potential for reuse of purified effluents and means of disposal of the concentrated wastes produced by the membrane process.
3. To demonstrate that the estimated process economics, based on non-cellulosic membrane materials, will be attractive in comparison with other color abatement processes available to the paper industry.

ULTRAFILTRATION

Ultrafiltration is a membrane process for concentration of dissolved materials in aqueous solution. A semi-permeable membrane is used as the separating agent and pressure as the driving force. In an ultrafiltration process, a feed solution is fed into the membrane unit, where water and certain solutes pass through the membrane under an applied hydrostatic pressure (permeate). Solutes whose sizes are larger than the pore size of the membrane are retained and concentrated (concentrate or retentate).

The pore structure of the membrane acts as a molecular filter, passing some of the smaller solutes and retaining the larger solutes. The pore structure of this molecular filter is such that it does not plug since larger solutes are rejected at the surface and do not penetrate the membrane. Thus, concentration of specific solution components can be achieved.

Considerations important for determining the technical and economic feasibility of ultrafiltration as applied are the rate of solution transport through the membrane (flux) and the separation efficiency (rejection). Other important factors include the membrane fouling rate, membrane cleanability, membrane material of construction and its physical properties and membrane geometry.

Ultrafiltration membranes that withstand almost any aqueous application are now being produced or developed in the membrane industry (33). This is significant in application of ultrafiltration to the process wastes under discussion since the streams treated in this study are hot (38 to 60°C) and have high pH (10-13). The availability of a membrane material which is stable at these conditions can reduce the pretreatment costs for the ultrafiltration processes.

Choice of the specific membrane material for use with pulp mill waste is complex but, in general, is based on membrane pH and temperature stability; controllability of pore size, to provide desired flux and rejection; fouling characteristics; cleanability and available geometric forms of modules.

At present, ultrafiltration membranes are available in five principle geometric configurations: spiral wound, tubular, plate and frame, hollow fiber, and dynamic membranes. The spiral wound unit is a double membrane sheath which is wrapped around a central permeate removal tube. Various types of separators (spacers) are in use to separate the membrane sheet to allow for concentrate flow through the spiral in a longitudinal direction.

A tubular unit consists of a porous tubular substrate which is cast coated, usually on the inside, with the membrane material.

The plate and frame unit is similar in construction to the traditional plate and frame filter. Effluent is pumped into a thin space between membrane covered plates continuously. Permeate is removed from the plates and concentrate from the space between plates.

Hollow fiber units consist of bundles of small diameter membrane tubes, hence providing large membrane area per cubic foot of membrane module volume. Flow of the material to be ultrafiltered is usually on the inside of the tubes. Permeate is removed from the outside of the tubes.

The dynamic membrane differs from the previous geometries in that the membrane is formed on a porous substrate by coating from suspension with a hydrous oxide.

The choice of the membrane geometry is significant in determining both the capital and operating cost of an ultrafiltration process. The advantages and disadvantages of membrane configurations have been discussed elsewhere (34). A review of cost-performance data has shown that spiral wound membranes hold promise of being lowest in costs. Some process limitations exist for membranes in this configuration. For this application, the potential economic advantages make it desirable to learn how to design this configuration so that it is adequate for processing both pine caustic extraction filtrate and decker effluents.

Membranes of porosity similar to Kodak HT-00 have been found to yield rejections in the 90-94% range for caustic extract, and 95-98% range for the decker effluents.

WASTE STREAM CHARACTERISTICS (NORTH CAROLINA MILL)

The waste streams which have been examined are the pine bleachery caustic extraction filtrate and the pine and hardwood pulp washing decker effluents. These streams are all highly colored. At the Canton mill, the caustic filtrate has an average color of about 20,000 c.u. (Color Units: basis Pt-Co Standard), the pine decker about 6,000 c.u. and the hardwood decker about 11,000 c.u.

Color in the deckers is predominately lignin materials which have been dissolved from the wood matrix in the pulping operation and has molecular weights predominately in the 4,000 to 8,000 range. Color in the caustic filtrate is due primarily to the shards of lignin and chlorinated lignin produced through oxidation in the bleaching operation. These materials appear to be predominately in the range of 400 to 4,000 molecular weight.

The pH's of the decker effluents are in the range of 10 to 11. The caustic filtrate pH is 11 to 12. The temperature of all three effluents are in the range of 38 to 60°C (100° to 140°F).

The suspended solids content of the caustic extract varies from about 50 ppm to 500 ppm. Suspended solids content of the pine decker is 50 to 250 ppm and for the hardwood decker, it is 100 to 500 ppm. The suspended solids content for all three streams is similar in that there are included varying amounts of pulp and clay materials. About one-half of the suspended solids are less than 10 microns in size.

There is another similarity in the solids content of these streams. Each stream contains perhaps 100 ppm of a micellar polysaccharide material which, under adverse conditions, can agglomerate and appear as a slimy foulant on the surface of membranes. This type of fouling can substantially reduce the membrane flux and necessitates membrane cleaning operations.

In the past several years, as a result of the intensive pulp and paper mill water conservation programs, this potential micellar fouling problem has been aggravated. The North Carolina mill has traditionally operated on an average of ten uses of water prior to discharge. It is now approaching twelve uses of water. To accomplish this, there is increased replacement of fresh water makeup for various operations by reuse of streams such as paper machine white water. These streams introduce additional micellar species such as starch and sizings which intensify the potential fouling problem.

The total solids in the streams vary but, in general, are one-quarter to one-third color body materials. The caustic extract has on average about 7,000 ppm of total solids with the predominant ions being sodium, calcium, hydroxide, chloride and sulfate. The pine decker has an average total solids of about 2,400 ppm and the hardwood decker about 3,000 ppm. In ultrafiltration of these streams, the multivalent ions such as aluminum and sulfate are usually rejected to some small extent. However, this rejection concentration is small enough that even at 100 times concentration of these streams, the osmotic pressure increase is small and it does not interfere with the low pressure requirements for membrane flux maintenance.

ULTRAFILTRATION PROCESS CONSIDERATIONS

The ultrafiltration processes for each of these pulp mill streams consists of a pretreatment system, a membrane system and a disposal system. The pretreatment system is designed to remove fibrous material and coarser suspended solids from the stream to be treated. A number of commercially available devices can be used for this purpose. With presently available membrane materials, neither stream pH or temperature (below 85°C) need to be controlled. Practical methods for eliminating slime forming material from these streams have not been demonstrated.

Technical feasibility has been demonstrated on a pilot scale for the use of pretreatment systems and membrane systems to remove color bodies by ultrafiltration from bleachery pine caustic extraction filtrate and pulp washing pine and hardwood decker effluents. The pilot scale demonstrations have been conducted at pressures of 3.4 to 6.8 atm (50 to 100 psig) with properly pretreated flow streams. Membrane flux levels of 0.82 to 3.28 m^3/m^2-day (20 to 80 gfd) have been demonstrated with color rejections in the 90-98% range at concentration ratios up to 100 times.

Use of ultrafiltration processes on decker effluents could permit closed cycle operation of the pulp washers with removal of its color contribution to total mill effluent. In addition, it would permit conservation of energy, salts, and water. Disposal of the concentrate from the decker effluents would be straightforward. Concentration to the proper level of organic material would allow for disposal of the concentrate in the weak black liquor streams. The inorganic chemical and organic material heat value recovery and reuse from black liquor are fundamental to kraft process economics. The permeate stream would be reused as make-up water in pulp processes.

Disposal of the concentrate from the caustic extraction filtrate is more complex because of the organic and inorganic chloride content. This concentrate may be disposed of by incineration in the lime kilns or with the black liquor, landfilled with lime sludge, or used for special chemical properties. The permeate can be recycled to the bleachery substantially, excess over this use can be used for low grade water on the mill site.

As described in detail in the following, the technical feasibility of color removal from kraft mill process effluents by ultrafiltration processes has been demonstrated. Projected costs and peripheral considerations of use of such processing in existing mills are attractive vis-a-vis alternative available technologies.

SECTION 2

CONCLUSIONS

This demonstration program has shown that ultrafiltration is a technically viable treatment process for color removal from kraft mill effluent streams. Processing takes place at natural stream temperature and pH levels due to the development of polysulfone (non-cellulosic) membrane formulations. Reuse of the ultrafiltered water (permeates) in pulping and bleaching operations appears practical and could provide water, material and energy conservation. The color concentrates (retentates) may be beneficially used (decker effluents) or treated in existing disposal systems (caustic filtration extract).

Tubular assemblies are the preferred ultrafiltration module geometry. This configuration exhibited high, stable flux performance, was readily cleanable and showed consistent color rejection. Spiral-wound cartridges, on the other hand, were plagued by severe fouling due to slime layer formation, had poor flux recovery upon cleaning and exhibited widely varying color rejection.

The economics of tubular ultrafiltration as projected, are cost-competitive with alternative treatment processes.

These general considerations are supplemented by the following specific findings:

1. FEED CHARACTERISTICS

   - Solids content, color, pH and temperature of the caustic extraction filtrate stream, pine decker effluent and hardwood decker effluent varied randomly from month-to-month. Wide fluctuations were observed on a daily basis. However, since ultrafiltration systems are generally insensitive to shock loading, and since field program data indicates no adverse effects from day-to-day feed variation no feed equalization is deemed necessary prior to processing.

   - Due to equipment changes, process changes or water conservation measures the nature of the three effluents has changed over the past four to five years. These types of changes can be expected to continue in the future but should pose little, if any problem for a tubular ultrafiltration system.

2. NON-CELLULOSIC MEMBRANE DEVELOPMENT

The conclusions presented in this subsection are based on laboratory studies of candidate membranes in flat sheet configuration. Laboratory studies are useful in screening membranes relative to each other with respect to various parameters. Data from such studies can be extrapolated to membranes in other geometric configurations.

- Polysulfone (WRP) membranes are preferred over specially coated type HFD and HFM membranes (commercially available from Abcor, Inc.) for this application. The best rejecting HFM membrane exhibited 98.5% rejection with an associated average flux of 0.86 m^3/m^2-day (5.1 atm) [21 gfd (75 psig)]. Two HFD membranes had 99% intrinsic color rejection but low flux. The majority of WRP membranes had color rejections of 99% to 99.5% with flux levels as high as 1.97 m^3/m^2-day (48 gfd).
- HFD and HFM membrane coating formulations degraded with time. This factor reinforced the preference for WRP-series membranes.
- High rejection-moderate flux ultrafiltration membranes can readily be made from polysulfone-based casting solutions. An entire series of membranes were cast from solutions containing different polymer concentrations with and without non-solvent, and with and without surfactant in the gelation bath. The preferred WRP membrane had a solvent:non-solvent: polymer weight ratio of 52.7:26.3:21.
- Interpolymer fixed charge membranes (prepared by H. Gregor, Columbia University) were compared with WRP membranes in both short and long term tests. The short term tests (3-hours) showed no practical differences between these membrane types. In long-term tests (65 hours) both membrane types exhibited essentially identical rates of flux decline. However, the WRP membranes showed higher color rejection and higher flux levels. Thus, the WRP membranes were selected for field evaluation.

3. FIELD EXPERIENCE WITH SPIRAL-WOUND MODULES

- Extensive processing of caustic extraction filtrate on a once-through basis was performed with a single module test stand. Sharp flux decline was consistently observed with 24-hour flux levels in the 0.12 to 0.41 m^3/m^2-day (3 to 10 gfd) range. Since no concentration occurred and since no increase in pressure drop was observed across the modules the flux decline pattern indicated a steady fouling of the membrane surface by some species in the caustic extraction filtrate. This foulant was subsequently identified to be a "slime" layer composed of volatile organics, kaolinite clay, starch, titanium dioxide and carboxylic acid salt. The major components of the fouling "slime" layer come from recycle of white water from the paper mill back to the pulp mill.

- Extensive testing in the areas of spiral-wound module pretreatment and prefiltration were ineffective in eliminating the foulants from the feed stream. Also, attempts to improve spiral-wound module flux performance by exploring a range of operating conditions proved unsuccessful. These results, coupled with significant flux recovery problems upon module cleaning, indicate that further spiral-wound module development will be required before this is a viable ultrafiltration membrane geometry for kraft mill effluent processing.

4. FIELD EXPERIENCE WITH TUBULAR ASSEMBLIES

- Polysulfone tubular assemblies in both 12.7 mm (0.5 in) and 25.4 mm (1 in) diameters exhibited high, stable flux performance during several hundred hours of caustic extraction filtrate processing. Operating on a once-through basis, flux for a 12.7 mm diameter tube (WRP formulation) stabilized at 1.64 to 2.26 m^3/m^2-day (40 to 55 gfd) for over 450 hours. No detergent cleaning or mechanical cleaning was necessary. 25.4 mm diameter tubular assemblies (commercial formulation) operated on caustic extraction filtrate at 3 concentration levels averaged these flux levels:

Concentration factor	Conversion	Average m^3/m^2-day (gfd)
1.2X	16.7%	2.87 (70)
10X	90%	2.26 (55)
50X	98%	1.03 (25)

Again, flux was stable over hundreds of operating hours. Flux recovery was rapid with a dilute detergent wash. The improved flux performance observed with tubular assemblies as compared to spiral-wound modules is a function of the higher superficial velocity over the membrane surface achievable with the tubular configuration. This leads to more turbulent flow and minimizes the gel ("slime") concentration layer at the membrane/liquid interface.

- Color removal by the tubular assemblies was exceptional, averaging 97% to 99% on an individual stage basis. Overall, on a mass discharge basis (kg color removed per day), 91% color removal is projected for processing of the caustic extraction filtrate stream to a 50X concentration.

5. PROJECTED ECONOMICS FOR FULL-SCALE SYSTEMS

- Eight full-scale system design cases were analyzed in terms of both capital and operating costs. For tubular systems treating 3,790 and 7,580 m^3/day (1 and 2 MM gpd) of either caustic extraction filtrate or decker effluent total capital investment ranges from $2 to 4 MM. Treatment costs, based on 727 metric tons of pine pulp produced per day (800 tons/day), range from $1.51 to $2.52 per metric ton ($1.36 to $2.27 per ton) for caustic extraction filtrate. The treatment costs range from $0.76 to $1.26 per metric ton ($0.68 to $1.13 per ton) for decker effluents on a 1,318 metric ton/day (1,450 ton/day) basis.

- Future technological advances are expected to reduce large-scale tubular ultrafiltraton system capital costs. Using future cost estimates, a 10% cost savings over current capital cost projections was calculated. The uncertainties associated with the future costs of labor, materials and equipment may, however, reduce this projected savings.

- Economic projections for spiral-wound module ultrafiltration systems were based on idealized systems not attainable with today's technology. Using idealized design flux values capital investments of $1 to $2.1 MM are projected. Treatment costs are $1.09 to $1.89/metric ton ($0.99 to $1.72/ton) of pulp for caustic extraction filtrate processing and $0.55 to $1.89/metric ton ($0.50 to $0.87/ton) of pulp for decker effluent processing.

6. WATER REUSE POTENTIAL

- The permeate from the ultrafiltration unit treating caustic extraction filtrate will constitute about 98% of the feed stream. This effluent will have low color, essentially no suspended solids and will have very low heavy metal content. In addition the permeate will have a high pH and be at process temperature.

  The permeate, with its physical and chemical attributes, should be an adequate water makeup stream for use in the bleachery processes. The high pH, and reduced buffering capacity should allow for lower new caustic requirements. The high temperature should reduce the system energy requirements. Because of the absence of suspended solids and the decreased heavy metal content, the permeate should reduce spray head and other scaling problems.

  It is believed that at least half of the permeate can be used in a bleachery recycle mode and that the savings in chemicals, water and energy from such use will have beneficial effects in reducing the net cost of the operation. Permeate which is excess can be admixed with mill input fresh water without discernable effects upon the fresh water quality. This is because the small permeate volume would be diluted 25 to 50 times by the larger fresh water input.

- The concentrate from ultrafiltration of caustic extraction filtrate has no direct value in pulp mill operations. Disposal of this material would be site-specific. In those installations equipped to remove chlorides from black liquor systems this concentrate could be flowed to the weak black liquor system with some small gain in energy recovery. Alternatively, the concentrate could be combusted in a typical modern lime kiln without noticeable effect, especially because of the low sodium content. Some mills which do not have sufficient lime kiln capacity dispose of the excess lime sludge off-site. Because of the high pH of this sludge and the relatively small volume of the concentrate, the concentrate could be added to the lime sludge and carried to landfill as insolubilized calcium salts.
- For ultrafiltration of pine and hardwood pulp washing decker effluents both permeate recycle to the pulping system for makeup water and concentrate recycle to the weak black liquor system are projected with concomittant cost reduction due to chemical, water and energy recovery values.

7. COMMERCIAL RELIABILITY

Further information is needed in several areas to establish the commercial reliability of the process.

- A larger scale process demonstration involving substantial numbers of modules and long term continuous operation is necessary to assess the manufacturer's capability to reproduce modules with requisite characteristics, and also, to acquire statistical data on module durability and useful life.
- Quantities of permeate and concentrate are needed to demonstrate reuse or disposal of these streams on a reasonable scale.
- The design and operation concepts used in this study were simplified for demonstration of the process on a limited scale. Validation of the process system projections is necessary using a prototype installation which models a full scale configuration, controls, operating protocols, cleaning systems, disposal systems, shock loading, etc. al.
- Operation of a prototype demonstration plant could provide information needed to "harden" the present projections of capital and operating costs, space requirements, operating and maintenance manpower, disposal of permeates and concentrates, shock response and the host of other considerations on which expanded data is needed to firm the concepts of commercial reliability.

SECTION 3

RECOMMENDATIONS

The major recommendation resulting from this study is field demonstration of tubular polysulfone membrane treatment of kraft mill effluent streams on a significant scale. A 190 to 379 m^3/day (50,000 to 100,000 gpd) staged pilot unit should be installed at a pulp mill and operated for at least 12 months. Circulation flowrate and inlet pressure requirements for each stage should be optimized to reduce overall system power consumption. Cleaning frequency and duration should be detailed and membrane life (mechanical failure, flux recovery with time) carefully monitored.

Full-scale system control and monitoring requirements should be considered as part of this demonstration and innovative engineering techniques for tubular ultrafiltration systems of this magnitude should be explored. In addition, the conceptual design and economic projections presented in this study should be updated.

Product streams, both permeate and concentrate, should be recycled within the mill to verify their projected reusability. Detailed chemical analyses for material balance studies should be continued.

Additional, more basic recommendations are:

Study of tubular module design to produce lower cost, more compact systems. For systems of the magniture required for kraft mill effluent streams - 3,790 to 7,580 m/day (1 to 2 MM gpd) - significant cost savings may be realized.

Spiral-wound module development should parallel tubular module design studies. Improvement in spiral-wound module feed-side spacer designs could increase turbulent flow and reduce (or eliminate) slime layer build-up. In such a case lower-cost, more compact ultrafiltration treatment systems would become available.

SECTION 4

PROGRAM OVERVIEW

The previous EPA sponsored documentation study report (32) on the use of ultrafiltration processes to control kraft mill effluent color presented a number of recommendations for subsequent work. In that study the technical feasibility and economic promise of such processes was demonstrated. The major recommendations emphasized the need for studies to improve and verify the economic bases for the process as new membrane capabilities were developed.

Studies conducted subsequent to 1973 demonstrated that the development of non-cellulosic membranes by the industry was at a point that promising non-cellulosic membranes were becoming commercially available and would be of value in this application.

With the support of EPA this program was undertaken as described in the foregoing.

The levels on which the program planning was done were principally:

1. Non-cellulosic membranes were commercially available which could operate with long life and provide:
   - operation at process stream temperatures and pH thus reducing costs of acid and cooling for pretreatment and also reducing the potential for stream micellar agglomeration to foul.
   - operation at fluxes 2 to 4 times those obtained with previously used cellulose acetate membranes thus providing the basis for a plant of given size flow capacity at a small fraction of the required membrane area. Thus, even though the cost per unit area of the new membrane was high, the capital cost of the plant would be maintained at that projected in the previous estimates despite inflation.
2. The commercially available non-cellulosic membranes could be available in spiral wound configurations with open channel spacers which would allow:
   - low cost per unit area membrane modules.
   - low pumping energy requirements compared to other membrane configurations.

- reduction of fouling due to higher superficial velocity over the membrane surface.
- ease of cleaning.

The program was initiated with a laboratory screening of a number of non-cellulosic membrane systems which could be commercially available. It was decided to primarily study the pine bleachery caustic extraction filtrate as the feed to be processed because it constitutes the major color contributor in a kraft pulp mill system and, also, because all previous studies have taught that the pulp washing decker effluents performed in a similar manner as regards membrane flux but with even more effective color rejection.

A commercially available membrane system was selected and demonstrated in the laboratory. When the system was installed in the mill using a "live" feed, however, severe fouling problems were observed.

It was determined that these fouling problems were a result of two interrelated factors:

1. As is increasingly true of most mills, in the interests of water conservation the Canton mill was using increasing amounts of "dirty" water (paper machine white water) for replacing fresh water makeup in the pulping and bleaching system. This introduced a greater amount of fouling material which the pretreatment system, as defined, did not remove.
2. Open spacer spiral-wound modules were not commercially available and the available Vexar spacer spiral-wound modules tended to build heavy slime-like polarized films that rapidly reduced the effective membrane flux.

Because of the very attractive economic advantages inherent in the use of spiral-wound membrane systems it was decided to study the possible methods of reducing the foulant potential of the feed streams to a level where the available spiral-wound membranes could operate effectively. Extensive studies were undertaken on available mechanical filtration equipment singly and in combination for this purpose. Studies were also conducted on combinations of these equipments together with a number of natural and synthetic flocculants and agglomerants. It was concluded after months of study that no practical solution would be available, within the time constraints of this program, which would permit economic control of this fouling potential in the mill stream.

The remainder of the program was devoted to demonstration of the potential of these non-cellulosic membranes in tubular form (commercially available) for kraft mill effluent color removal. The effectiveness of this geometric form of the selected membranes was demonstrated to be practical both technically and on the basis of the projected economics.

The following report sections present discussions of the scope and details of this study.

SECTION 5

EXPERIMENTAL EQUIPMENT AND PROCEDURES

PILOT PLANT

General

The pilot plant for kraft pulp mill effluent treatment studies was designed as a 3.79 m^3/day (10,000 gpd), three-stage system utilizing Vexar spacer spiral-wound membrane modules. A simplified system flow schematic for the pilot plant is shown in Figure 1. The pilot system contained two major areas: a pretreatment and prefiltration section and a 3-stage ultrafiltration section. Photographs of various system components are shown in Figures 2 through 10. The operation of these components is discussed below.

Pretreatment and Prefiltration Section

The pretreatment and prefiltration section of the pilot plant (see Figure 11) was designed to remove suspended solids from the feed streams. Suspended solids (including fibers) reduction was necessary to prevent plugging and reduce fouling of the spiral-wound membrane modules. Pretreatment (investigated during a portion of the field program only) consisted of polymer addition to the feed stream to flocculate suspended particles and aid in their removal by the system prefilters. Components of the pretreatment system were a polymer solution tank, a Milton-Roy metering pump, a mixing tank, and a Hoffman Vac-20 Vacu-matic filter. The polymer solution was pumped by the metering pump to the mixing tank where it was combined with the feed stream. Flow from the mixing tank to the Hoffman filter was by gravity. The Hoffman filter contained a pump to transfer the filtrate to the system feed tank.

The Hoffman Vac-20 Vacu-matic filter is a flat-bed vacuum filter. It consists of an endless metal conveyor belt which supports the filter's media and passes it over a vacuum chamber. During filtration, as the filter cake increases in thickness and filtrate flow decreases, the Vac-20 automatically indexes fresh filter media. The filter media are supplied in roll form and are available with a wide range of effective pore sizes.

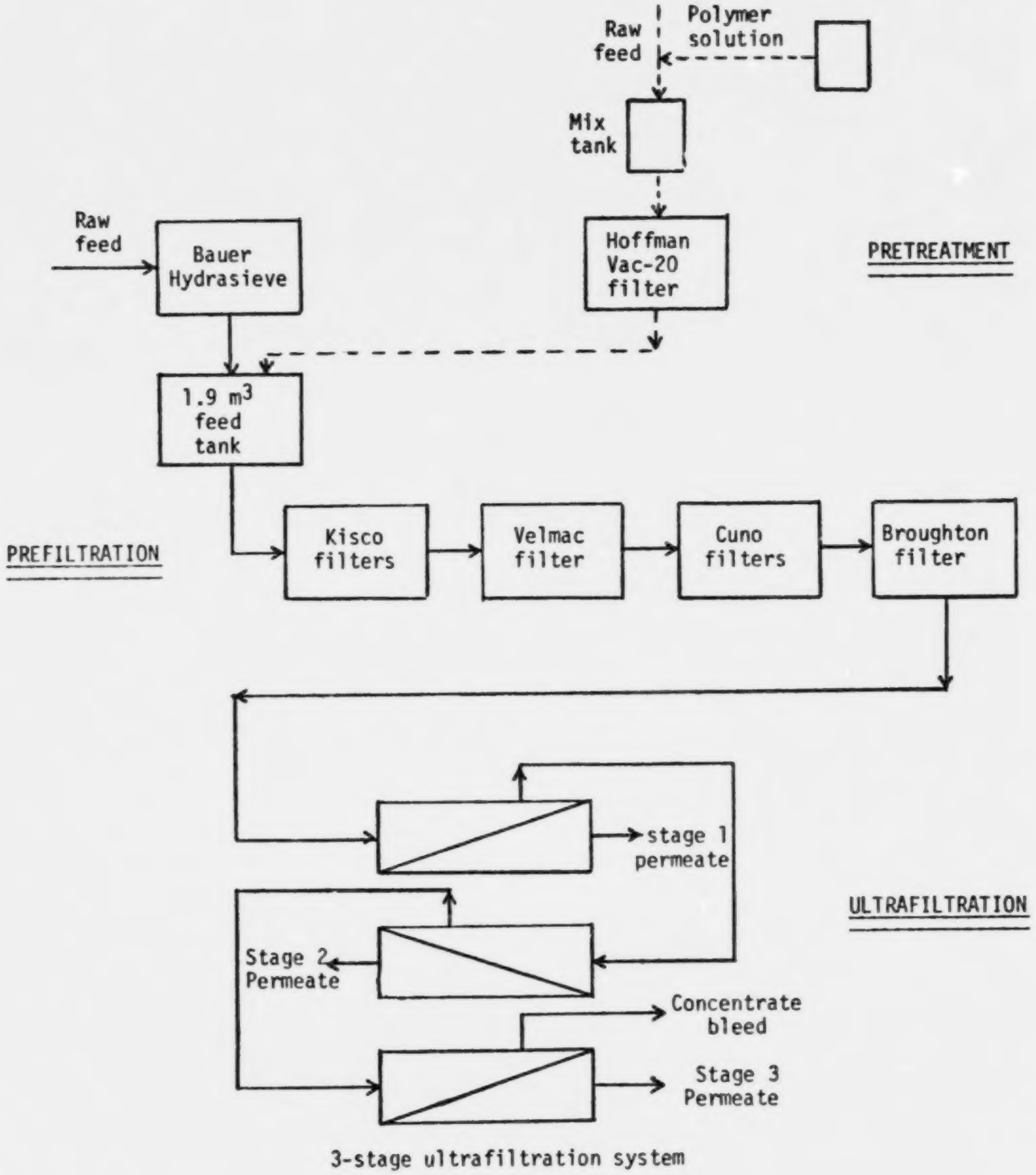
Raw feed
Polymer solution
Mix tank
Raw feed
Bauer Hydrasieve
Hoffman Vac-20 filter
PRETREATMENT
1.9 m3 feed tank
PREFILTRATION
Kisco filters
Velmac filter
Cuno filters
Broughton filter
stage 1 permeate
ULTRAFILTRATION
Stage 2 Permeate
Concentrate bleed
Stage 3 Permeate
3-stage ultrafiltration system

Figure 1. Simplified flow schematic of Canton Mill pilot system.

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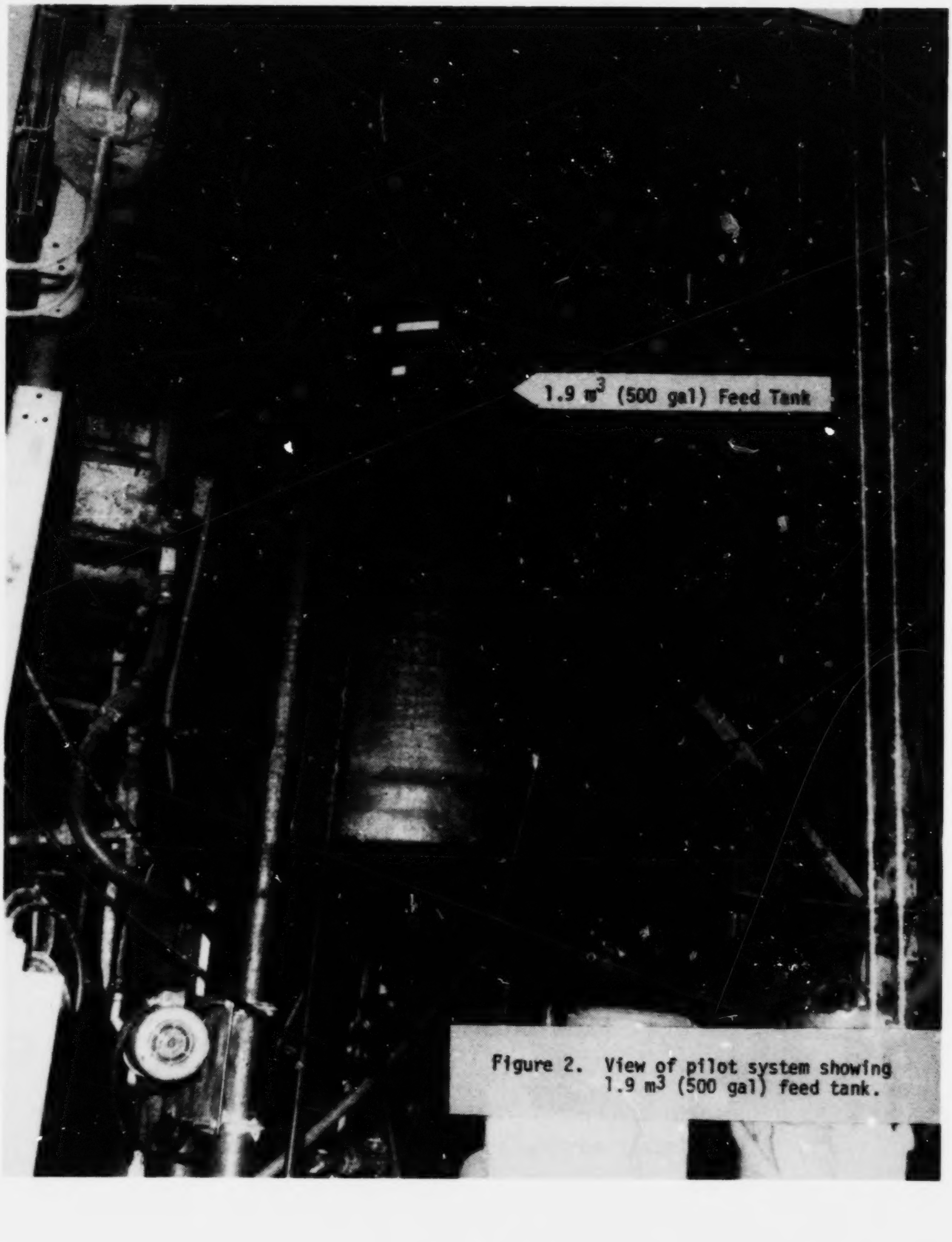
1.9 m^3 (500 gal) Feed Tank

Figure 2. View of pilot system showing 1.9 m^3 (500 gal) feed tank.

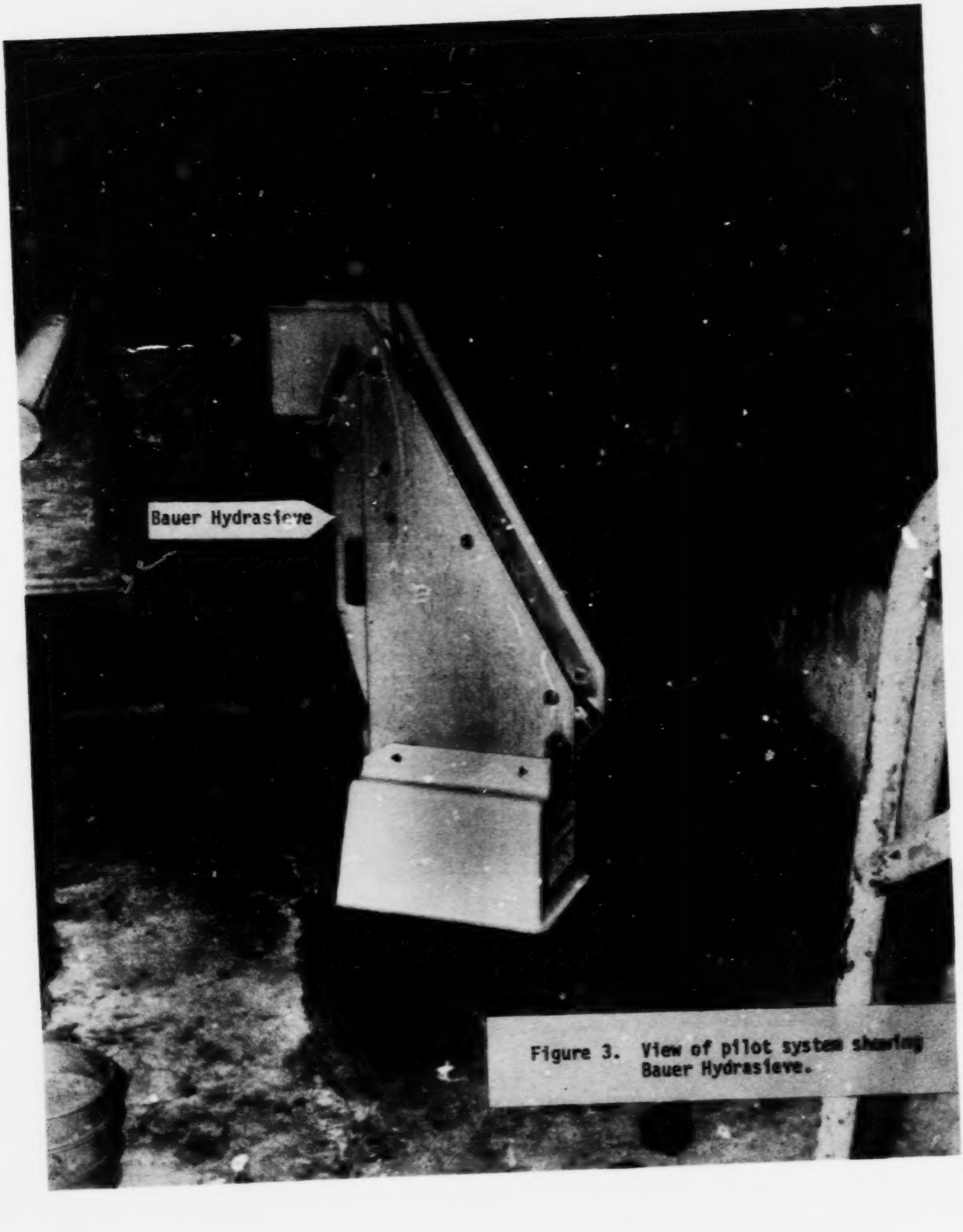
Bauer Hydrasieve

Figure 3. View of pilot system showing Bauer Hydrasieve.

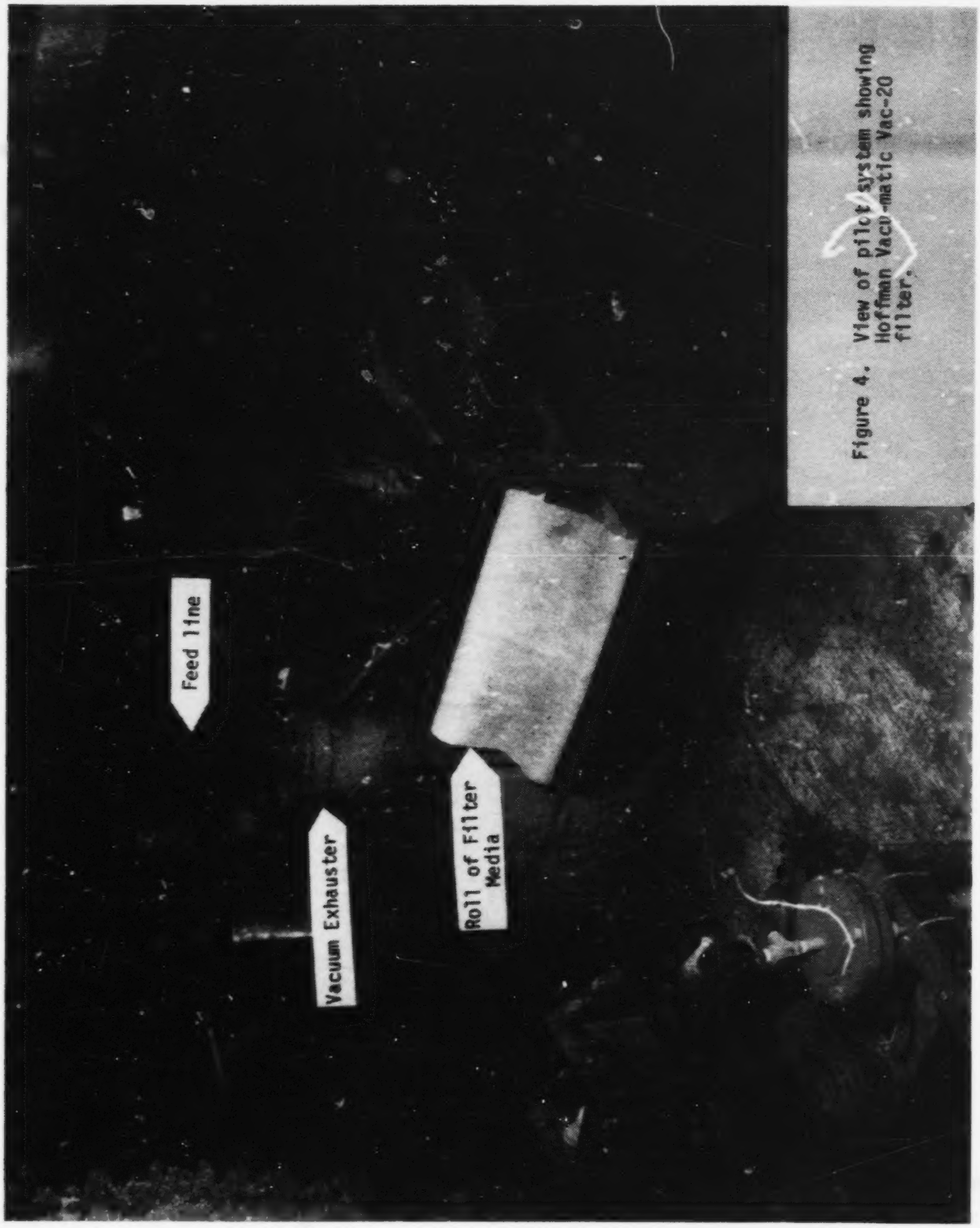
Feed line
Vacuum Exhauster
Roll of Filter Media

Figure 4. View of pilot system showing Hoffman Vacu-matic Vac-20 filter.

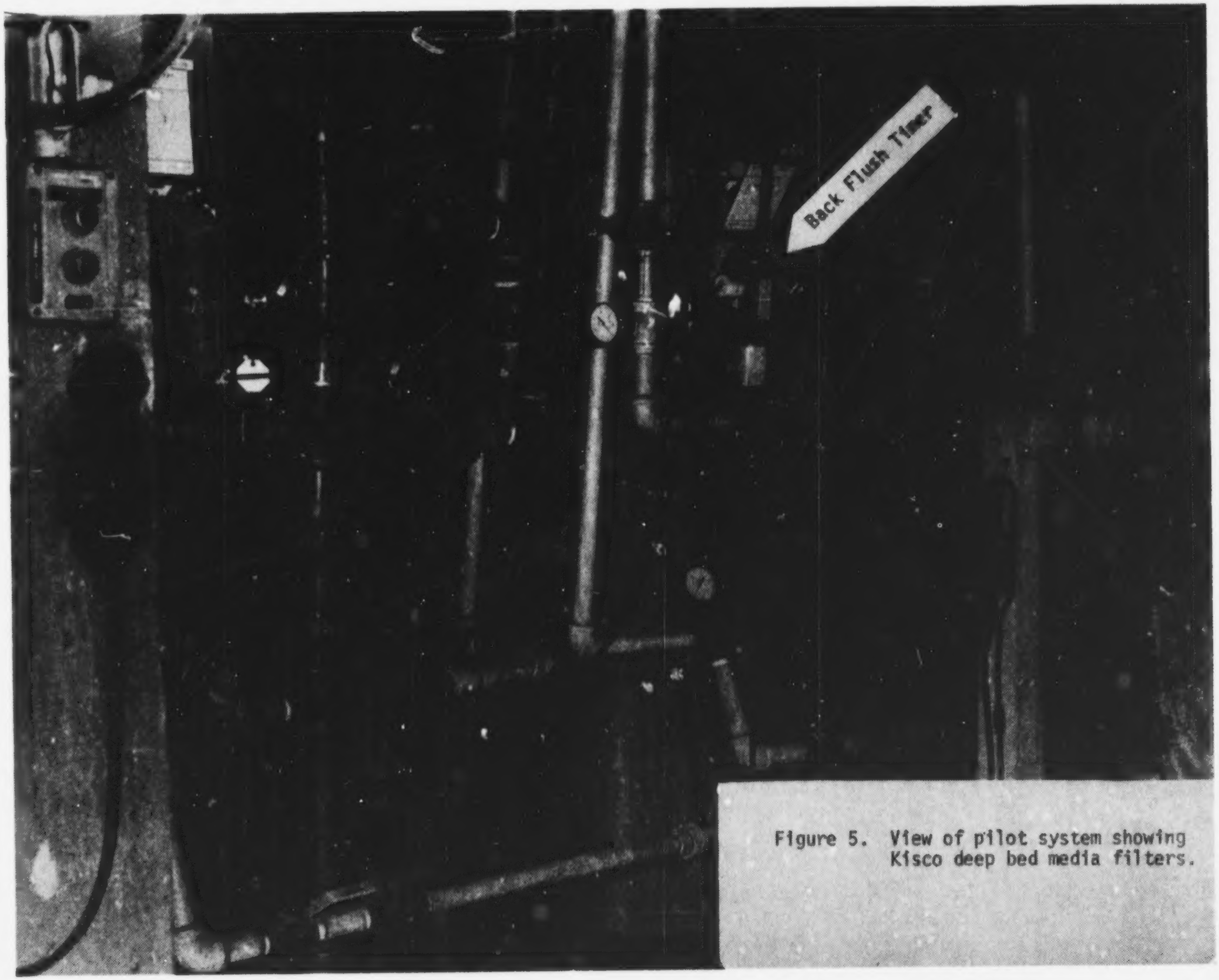
Back Flush Timer

Figure 5. View of pilot system showing Kisco deep bed media filters.

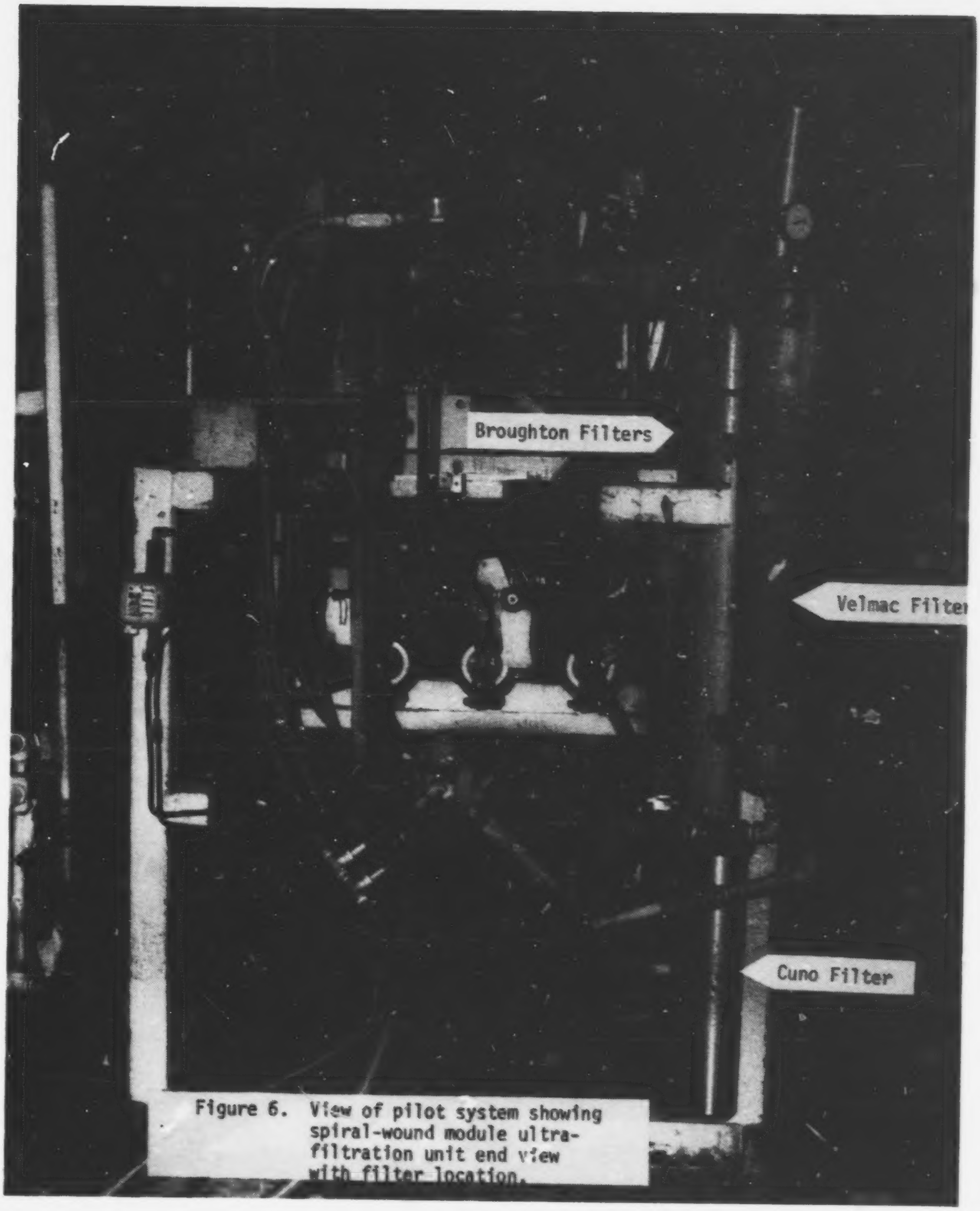
Broughton Filters
Velmac Filte
Cuno Filter

Figure 6. View of pilot system showing spiral-wound module ultrafiltration unit end view with filter location.

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best available copy.

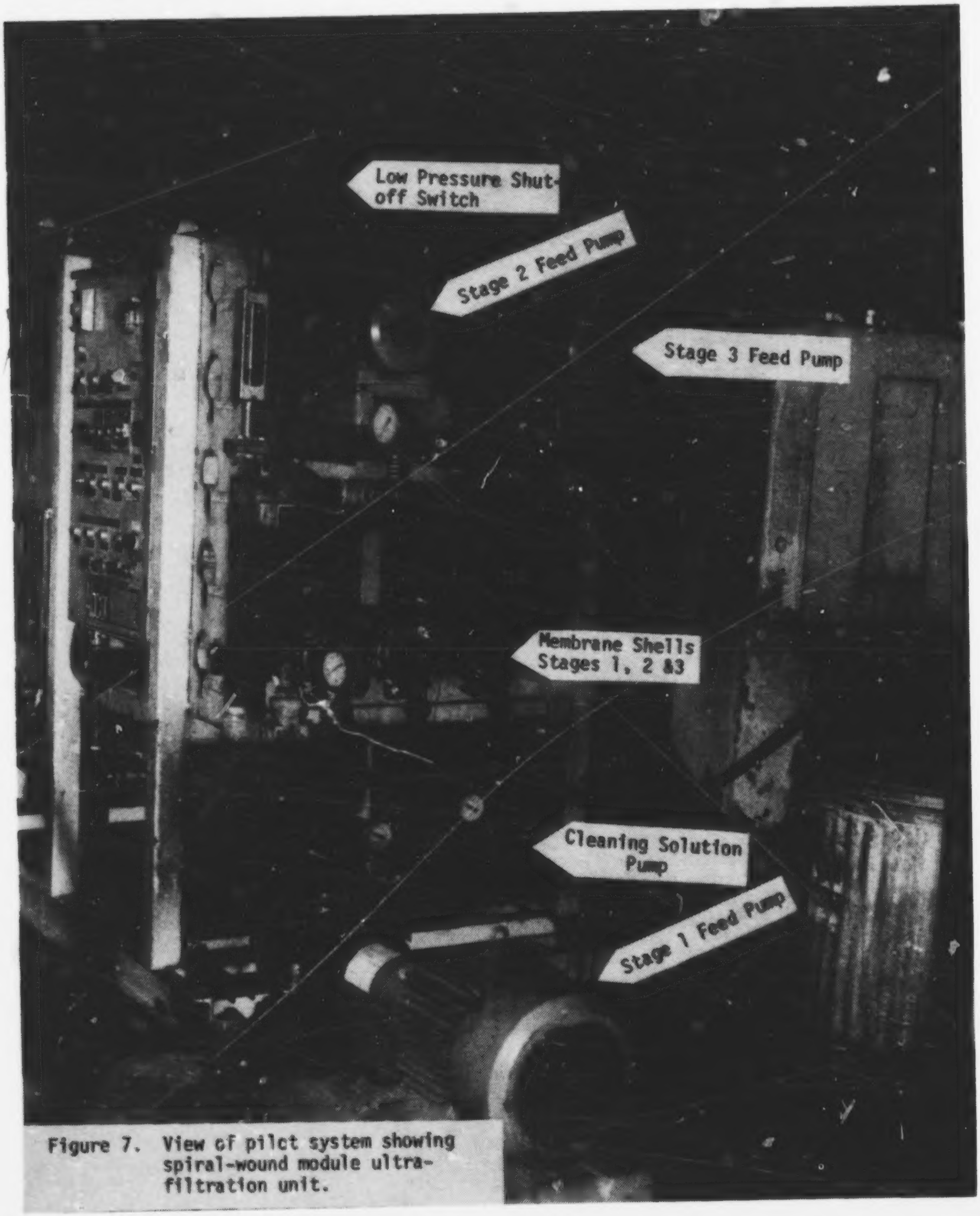
Low Pressure Shut-off Switch
Stage 2 Feed Pump
Stage 3 Feed Pump
Membrane Shells Stages 1, 2 &3
Cleaning Solution Pump
Stage 1 Feed Pump

Figure 7. View of pilot system showing spiral-wound module ultrafiltration unit.

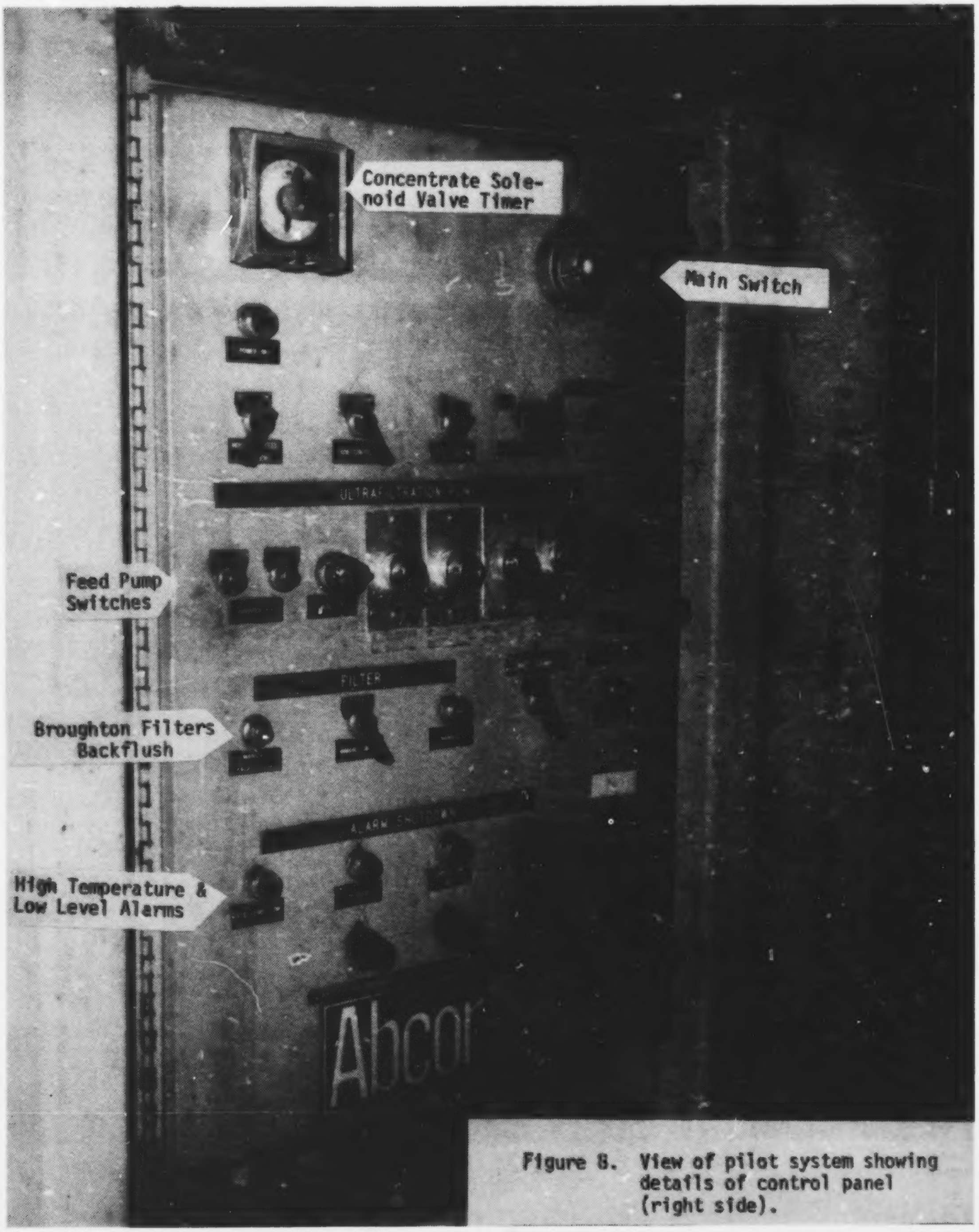
Concentrate Solenoid Valve Timer
Main Switch
ULTRAFILTRATION
Feed Pump Switches
FILTER
Broughton Filters Backflush
ALARM SHUTDOWN
High Temperature & Low Level Alarms
Abco

Figure 8. View of pilot system showing details of control panel (right side).

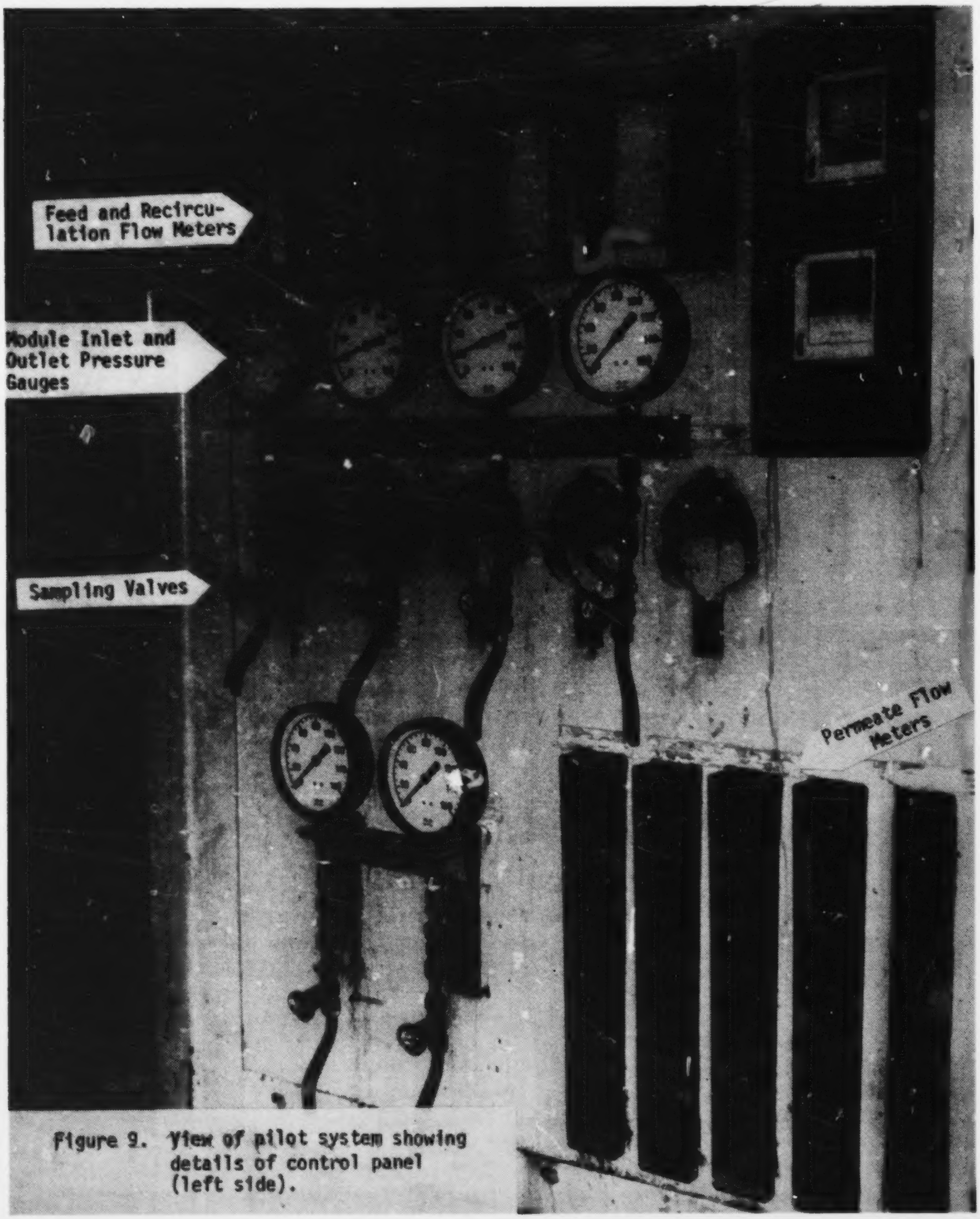
Feed and Recirculation Flow Meters
Module Inlet and Outlet Pressure Gauges
Sampling Valves
Permeate Flow Meters

Figure 9. View of pilot system showing details of control panel (left side).

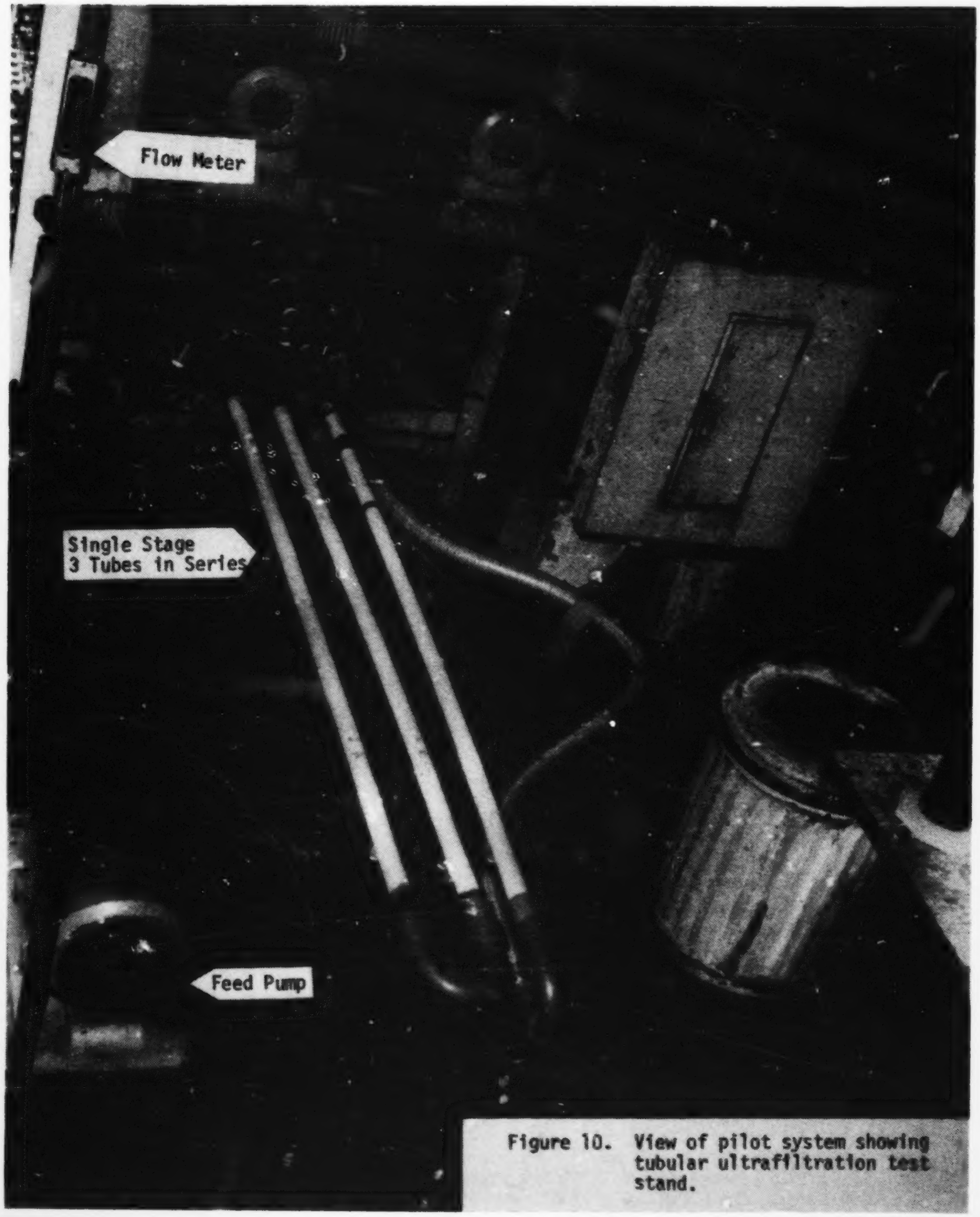
Flow Meter
Single Stage
3 Tubes in Series
Feed Pump

Figure 10. View of pilot system showing tubular ultrafiltration test stand.

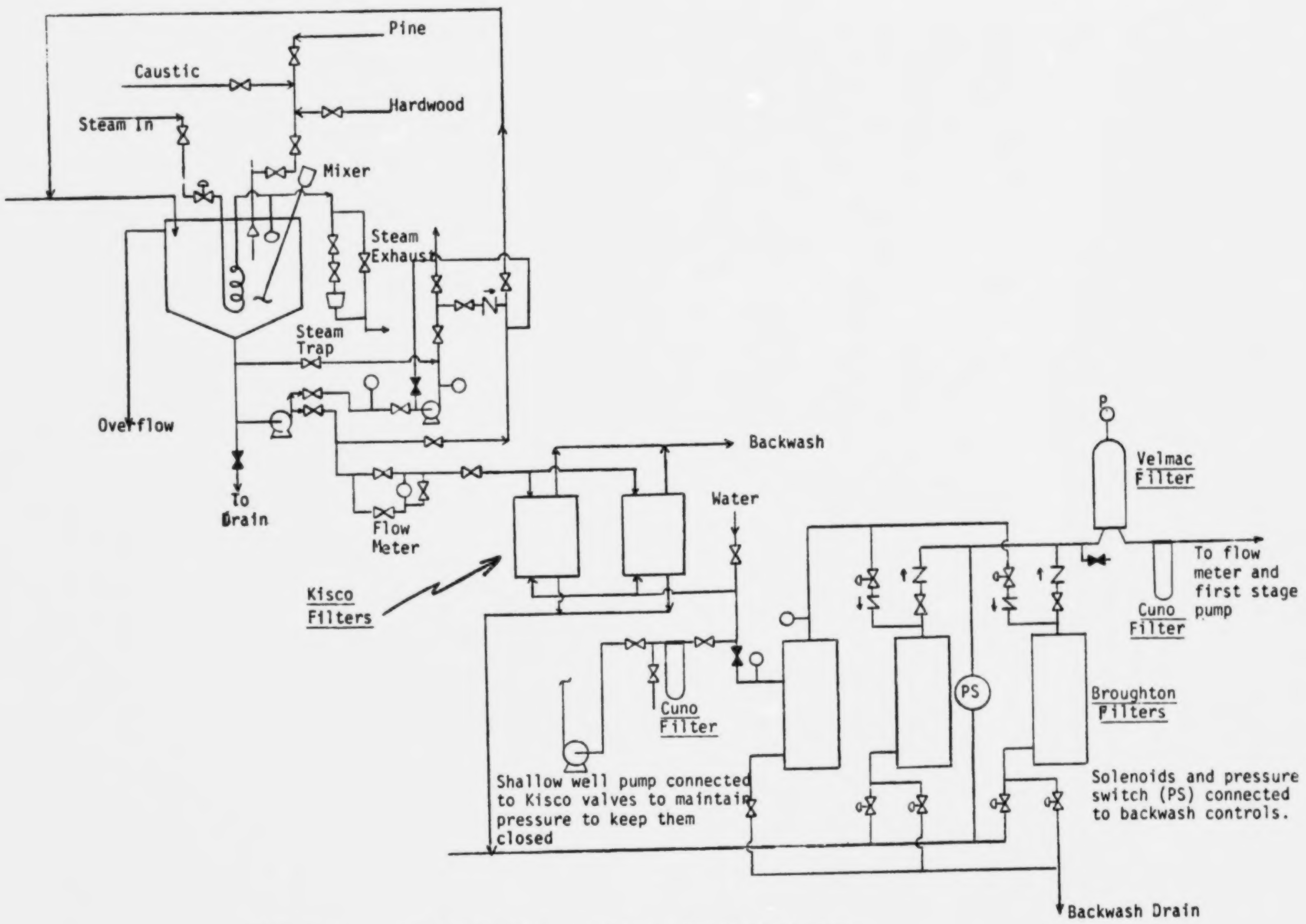
Pine
Caustic
Hardwood
Steam In
Mixer
Steam Exhaust
Steam Trap
Overflow
To Drain
Flow Meter
Backwash
Kisco Filters
Water
P
Velmac Filter
To flow meter and first stage pump
Cuno Filter
PS
Broughton Filters
Cuno Filter
Shallow well pump connected to Kisco valves to maintain pressure to keep them closed
Solenoids and pressure switch (PS) connected to backwash controls.
Backwash Drain

Figure 11. Prefiltration section of pilot system.

The equipment in the prefiltration area consisted of a Bauer Hydrasieve, a system feed tank, a Kisco deep bed media filter, a Broughton basket type filter, a Velmac disc filter, and a Cuno cartridge filter. The process flow pattern was designed to utilize these filters in series (in the order listed above). The Bauer Hydrasieve removed fibers from the feed stream and was located upstream of the system feed tank. The Hydrasieve was used for part of the program but was discontinued when Kisco personnel indicated that the fiber mat build-up on the Kisco media would aid in small suspended particle removal.

The 1.9 m^3 (500 gal) system feed tank was equipped with a level control, temperature control, and a mixer. Process stream temperature for the total system was controlled at this point. From the feed tank the process stream was pumped to the Kisco deep bed media filters. These filters contained a 0.76 m (30 in) bed of Filter AG (granular non-hydrous aluminum silicate) media. Two Kisco filters were piped in parallel; one filter on the process stream and one backwashing or on standby. The effluent from the Kisco filters was passed through the Broughton filters, the Velmac filter and the Cuno filter, in series. While the Broughton filters were used throughout the program, the Velmac and Cuno filters had only limited use. When not in use the Velmac and Cuno cartridges were removed from their housings. Following the Cuno filter the process flow entered the ultrafiltration section of the pilot system.

Ultrafiltration Section

The ultrafiltration section of the pilot system contained three recirculated stages in series. Each stage consisted of a module housing, valves, piping, and a pump. Two Vexar spacer spiral-wound membrane modules could be positioned in each housing. These 0.1 m (4 in) diameter x 0.9 m (36 in) long modules each contained approximately 3.7 m^2 (40 ft^2) of membrane area. Feed recirculation was required to maintain sufficient flow rates through the modules. Also, a single pass system would not give adequate process stream circulations (see Figure 12 for design flow rates).

The circulation pumps on each stage were piped with a recycle line, high and low pressure switches, and valves for individual stage control. The total flow through each stage, ratio of fresh feed to recirculated feed, and pressure to the stage were all controlled. The high pressure switches protected membrane modules from over pressurization. The low pressure switches shut the system down for pump protection in case of system upset. As stated earlier the feed tank in the prefiltration section had temperature control and a high temperature shut-off switch.

SINGLE MODULE TEST STAND

The single module test stand was designed to test pretreatment and prefiltration efficiency on the process feed stream and to evaluate module performance characteristics and cleaning requirements. The test module

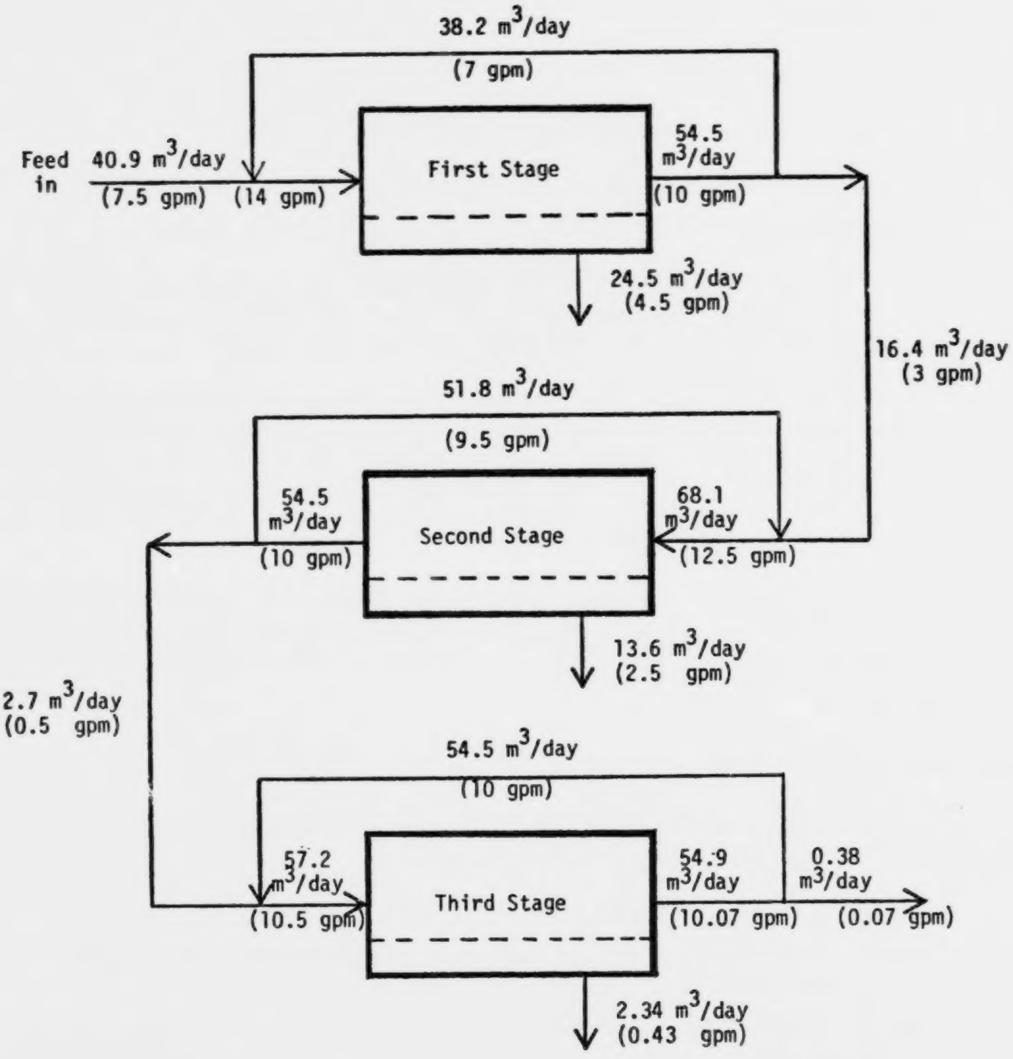
38.2 m³/day
(7 gpm)
Feed in
40.9 m³/day
(7.5 gpm)
(14 gpm)
First Stage
54.5 m³/day
(10 gpm)
24.5 m³/day
(4.5 gpm)
16.4 m³/day
(3 gpm)
51.8 m³/day
(9.5 gpm)
54.5 m³/day
(10 gpm)
Second Stage
68.1 m³/day
(12.5 gpm)
13.6 m³/day
(2.5 gpm)
2.7 m³/day
(0.5 gpm)
54.5 m³/day
(10 gpm)
57.2 m³/day
(10.5 gpm)
Third Stage
54.9 m³/day
(10.07 gpm)
0.38 m³/day
(0.07 gpm)
2.34 m³/day
(0.43 gpm)

Figure 12. Design flow rates for 3-stage ultrafiltration pilot system.

shell held a single 0.1 m (4 in) diameter spiral-wound module. Mill effluent was processed through the module on a once-through basis exposing the module to high volumes of fresh feed. The stage-1 pump was used as the feed pump for the test module stand. Temperature, pressure and flow rates similar to proposed pilot system operating conditions could be readily met with this system.

TUBULAR MODULE TEST STAND

The tubular module test stand served two functions:

- -- Determination of tubular membrane performance characteristics; and,
- -- Operation as a single stage recirculating pilot system.

To evaluate tubular membrane fouling properties the system was operated as a single pass system. The main pilot system prefiltration system was used and the Stage 1 pump was the feed pump for the test stand.

The single stage recirculating pilot system consisted of 2, 3, or 4 tubular membrane assemblies in series. Both 12.7 mm (0.5 in) diameter and 25.4 mm (1 in) diameter tubular assemblies were tested. The 12.7 mm (0.5 in) diameter tubes were tested in various lengths, with and without turbulence promoters. The 25.4 mm (1 in) diameter tubes were 1.5 m (5 ft) long and contained 0.1 m^2 (1.1 ft^2) of membrane surface area.

The tubular system was operated at 136 m^3/day (25 gpm) and 109 m^3/day (20 gpm) recirculation rates. At a 1.2X concentration this single stage tubular membrane test stand had a 27.3 m^3/day (7,200 gpd) capacity.

MEMBRANE CLEANING PROCEDURES

WRP Membrane Spiral-Wound Modules

The cleaning procedure for WRP membrane spiral-wound modules found to be most effective consisted of three steps:

Step 1: Flush the system with 49°C (120°F) water.

Step 2: Prolonged recirculation of a cleaning solution through the system.

Step 3: Flush the system with water.

A description of each step follows:

Step 1: The flushing of the system with 49°C (120°F) water was an important part of cleaning. The fouling layer was substantially removed by this warm water flush. When cold water was used, the fouling layer apparently set-up and was much harder to remove in Step 2. Typically, this warm water flush was done with a 1.7 atm (25 psig) pressure on the inlet side and 27.3 to 43.6 m^3/day (5 to 8 gpm) flow rate. The 49°C water was fed from the cleaning tank and sewered after going through the system. The operating procedure was to fill the 0.38 m^3 (100 gal) cleaning tank with water, heat to 49°C with live steam, and then pump through the system until the cleaning tank went dry. This gave a 10-15 minute water flush.

Step 2: The most effective cleaning composition was a solution of 0.5% NaOH and 0.25% EDTA. This cleaning solution was pumped through the system and totally recycled back to the cleaning tank. The normally used cleaning conditions were:

a) 1.7 to 3.4 atm (25 to 50 psig) inlet pressure
b) 43.6 to 65.4 m^3/day (8 to 12 gpm) recirculation rate
c) 49 to 54°C (120 to 130°F)
d) one hour duration

The cleaning system pump was incapable of pumping the 43.6 to 65.4 m^3/day (8 to 12 gpm) desired recirculation rate. The high recirculation rate was obtained by using the Stage 1 pump in series with the cleaning system pump.

Step 3: The cleaning solution was flushed from the system with warm water. Normally 49°C water was used, but cold water seemed to be just as effective. The final flush was to remove all the dirty cleaning solution from the system. The flushing water was pumped from the cleaning tank through the system and then sewered. Typically, this was done at 27.3 to 43.6 m^3/day (5 to 8 gpm) with an inlet pressure of 1.7 atm (25 psig).

After cleaning the membrane modules, a water flux was run. If the flux was less than 1.64 m^3/m^2-day (40 gfd) Steps 2 and 3 were repeated. A 1/2% solution of Ultraclean was usually effective at this point in the repeat of Step 2.

Appendix A contains a summary of cleaning materials used.

WRP Membrane Tubular Assemblies

An almost total lack of fouling problems in ∿1000 hours operation with the WRP membrane tubular assemblies resulted in little cleaning information for this membrane geometry. The cleaning procedure used was essentially the same as for the spiral wound modules:

Step 1: Flush system with 49°C (120°F) water.

Step 2: Recirculation cleaning with 0.5% Ultraclean detergent for two hours.

Step 3: Flush system with water.

In step 2, the system was recycled at 136 m^3/day (25 gpm) flow rate with a pressure of 3.4 atm (50 psig) and a temperature of 49°C. Recirculation time was two hours but probably was in excess of the time necessary to clean the membranes.

This three-step cleaning procedure resulted in water flux at 49°C of 5.33 to 6.97 m^3/m^2-day (130 to 170 gfd) for the clean WRP membrane tubular assemblies.

51 MM DIAMETER DEPTH FILTER

A 51 mm (2 in) diameter laboratory-scale depth filter was tested at the Canton Mill to assess the suspended solids removal efficiency of various filter media. The media evaluated were:

Media	Bed depth
- Anthracite coal	0.46 m (18 in)
Silica sand	0.25 m (10 in)
Garnet sand	0.05 m (2 in)
- Garnet sand	0.76 m (30 in)
- Filter Ag	0.76 m (30 in)
- Manganese green sand	0.76 m (30 in)
- Granular PVC	0.76 m (30 in)

A flow schematic of the depth filter test system is shown in Figure 13. A photograph of the depth filter system is shown in Figure 14. A slip stream of caustic extraction filtrate was fed to a 54.5 m^3/day (10 gpm) Bauer Hydrasieve to remove fiber. The Hydrasieve underflow was collected in a surge tank and transferred to the uppermost portion of the column by a metering pump. The column was constructed of translucent polycarbonate to allow visual inspection of the media. Surface caking and/or stream channeling could thus be observed and corrected.

The feed percolated through 0.76 m (30 in) of filtering medium. When anthracite coal and silica sand were employed the intermix zone was 82 mm (3.25 in). In all cases the column backflush expansion height was 0.17 m (6.7 in). The media was supported by a fine-mesh screen and a perforated plate used to evenly distribute the backwash flow. The inlet pressure to the filter bed and the filtrate suspended solids content were measured. The flow rate through the column was maintained at 29.4 m^3/m^2-day (5 gpm/ft^2). The feed solution was at actual temperature (52-57°C) and pH (pH = 11.5).

During regeneration, filtrate was fed through the base of the column, and the media bed was expanded to the full column height. The backwashing was typically performed for 5 to 8 minutes at a flow rate of 88 m^3/m^2-day (15 gpm/ft^2).

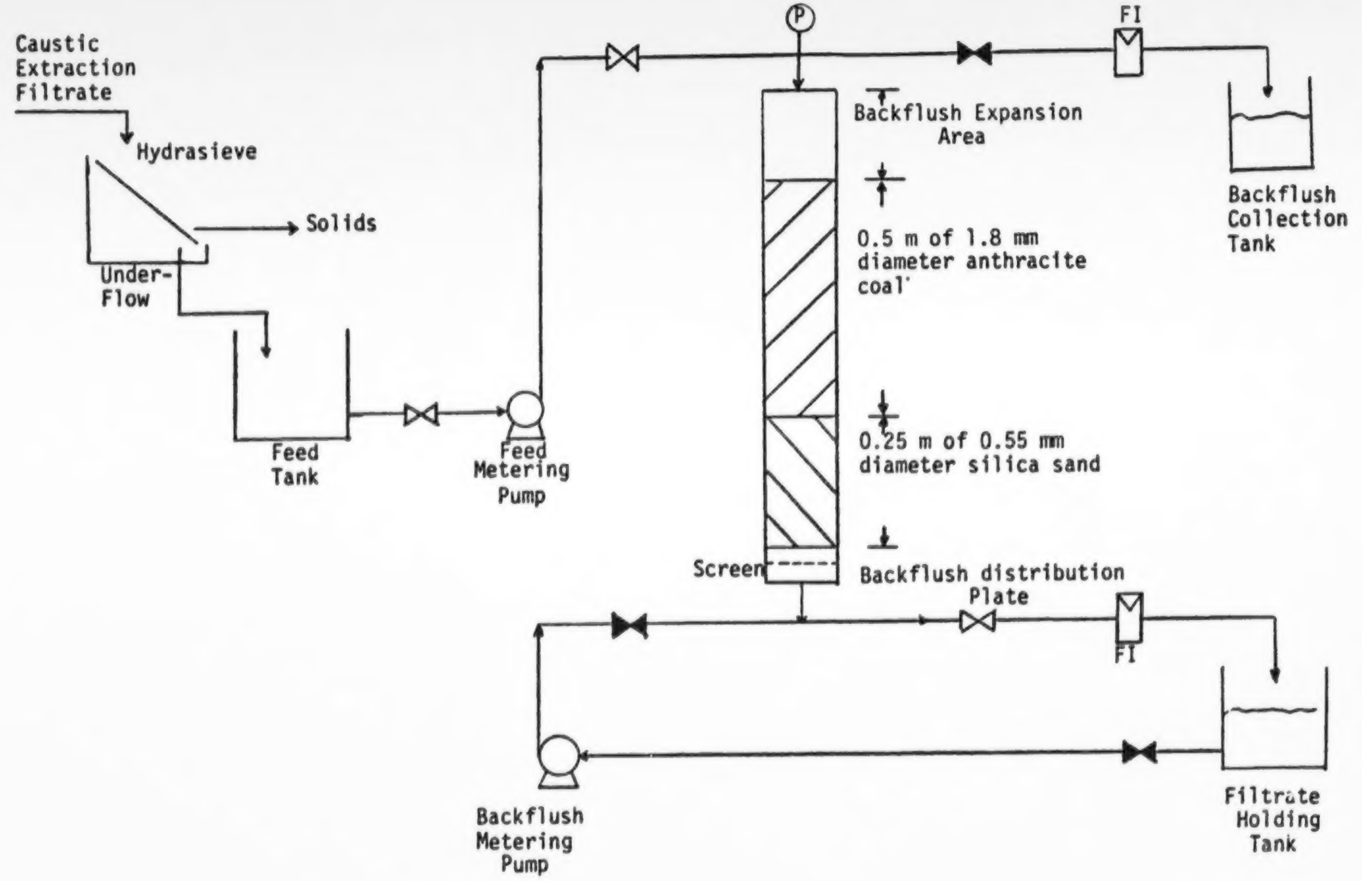
Caustic
Extraction
Filtrate
Hydrasieve
Solids
Under-
Flow
Feed
Tank
Feed
Metering
Pump
P
FI
Backflush Expansion
Area
0.5 m of 1.8 mm
diameter anthracite
coal
0.25 m of 0.55 mm
diameter silica sand
Screen
Backflush distribution
Plate
FI
Backflush
Collection
Tank
Filtrate
Holding
Tank
Backflush
Metering
Pump

Figure 13. Flow schematic for 0.05 m diameter depth filter test system.

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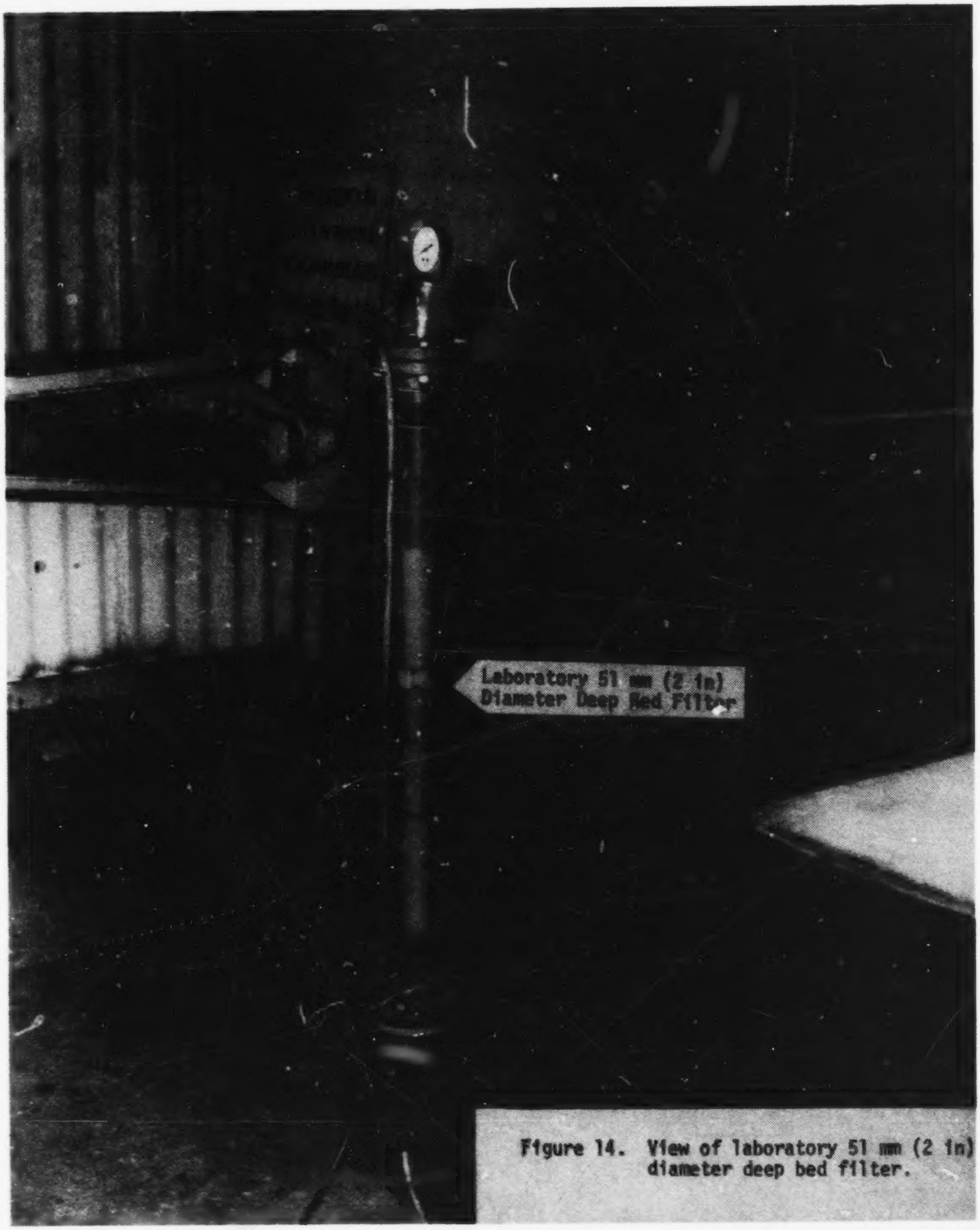

Laboratory 51 mm (2 in)
Diameter Deep Bed Filter

Figure 14. View of laboratory 51 mm (2 in) diameter deep bed filter.

LABORATORY ULTRAFILTRATION SYSTEM

A simplified flow diagram of the UF test system employed for tubular assembly parametric tests and module screening tests prior to mill evaluation is shown in Figure 15. A centrifugal booster pump (Dayton Model 6K507) was used to provide sufficient pressure to pass the feed through two 400 μ stainless-steel strainers, in parallel, for removal of gross solids. A centrifugal circulation pump (Worthington Model D-820) was used to pressurize the feed and pass it through the membrane module. The flow rate and pressure were controlled by the pump bypass valve (V-8) and the concentrate throttle valve (V-7). A low pressure switch (LPS) protected the pump from running dry. The concentrate could be recycled either to the feed tank or to the suction of the circulation pump. A temperature controller (United Electric, Type 1200) and heat exchanger were used to control the temperature at a predetermined level. The permeate and concentrate flow rates were measured, and the feed flow rate was calculated (sum of concentrate and permeate flows). The feed pressure and pressure drop across the module were also determined.

The test system shown in Figure 15 was typically operated in the total recycle mode. In this operating mode both the concentrate and permeate streams are returned to the feed tank and the feed is time-invariant.

STIRRED CELL ULTRAFILTRATION SYSTEM

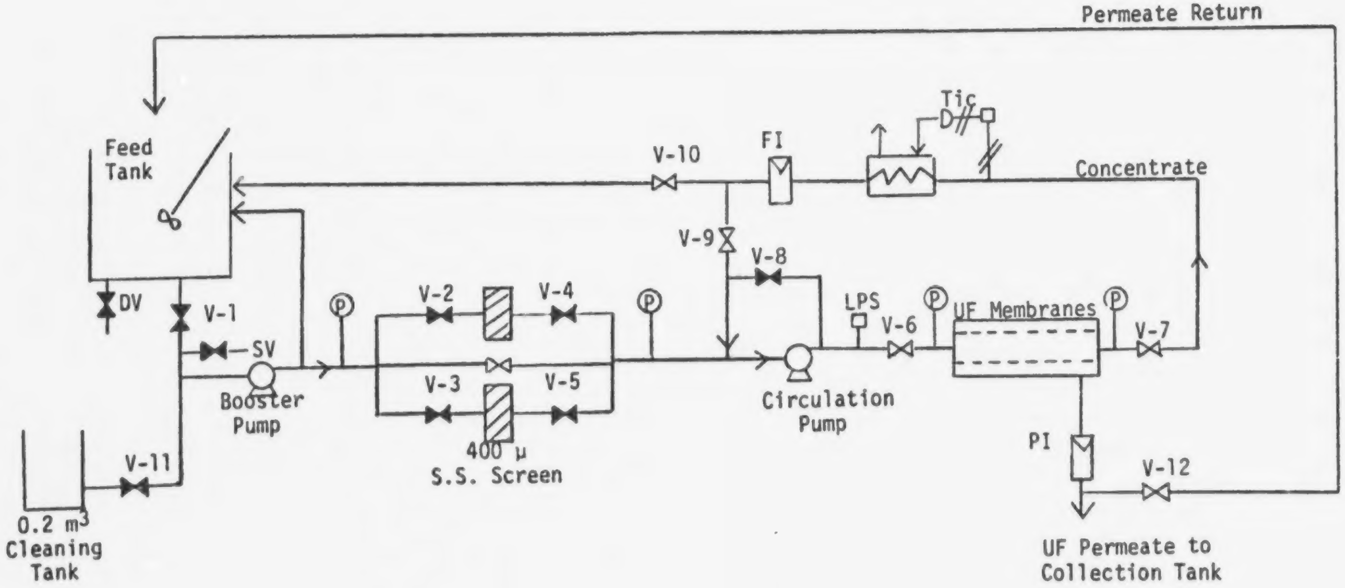
Single Cell Tests

The screening tests for evaluation of non-cellulosic membranes were performed in stirred cells operated as shown in Figure 16. The membrane to be tested was placed in the stirred cell (Amicon Model 202) and the cell was filled with 180 ml of caustic extraction filtrate at ambient temperature. The magnetic stirrer was then started and the cell was pressurized to 5.2 atm (75 psig). The first 50 ± 2 ml of permeate was collected and discarded while the second 50 ± 2 ml was collected and analyzed for color. A raw feed sample was also analyzed for color. During the test the volume processed with time was carefully monitored.

Following the test with caustic extraction filtrate the membrane was subjected to a salt rejection test under the same operating conditions using a solution of magnesium sulfate as feed. The first 10 ± 0.5 ml of permeate was discarded and the next 20 ± 1 ml collected and analyzed for conductivity.

Membranes were evaluated in terms of average flux, apparent color rejection, intrinsic color rejection and salt rejection. An explanation of each of these evaluation criteria follows:

- Average flux: The average flux was obtained by dividing the total volume permeated in the test (100 ml) by (1) the time required to process the permeate and (2) the total surface area of the flat sheet in the stirred cell (31 cm^2).


Permeate Return
Feed Tank
V-10
FI
Tic
Concentrate
V-9
V-8
DV
V-1
SV
Booster Pump
V-2
V-4
V-3
V-5
400 μ
S.S. Screen
LPS
V-6
UF Membranes
V-7
Circulation Pump
PI
V-12
V-11
0.2 m³ Cleaning Tank
UF Permeate to Collection Tank

Legend

DV - Drain Valve
FI - Flow Indicator
LPS - Low Pressure Switch
P - Pressure Indicator
SV - Sample Valve
TIC - Temperature Indicator/Controller

Figure 15. Simplified flow schematic of laboratory ultrafiltration test system.

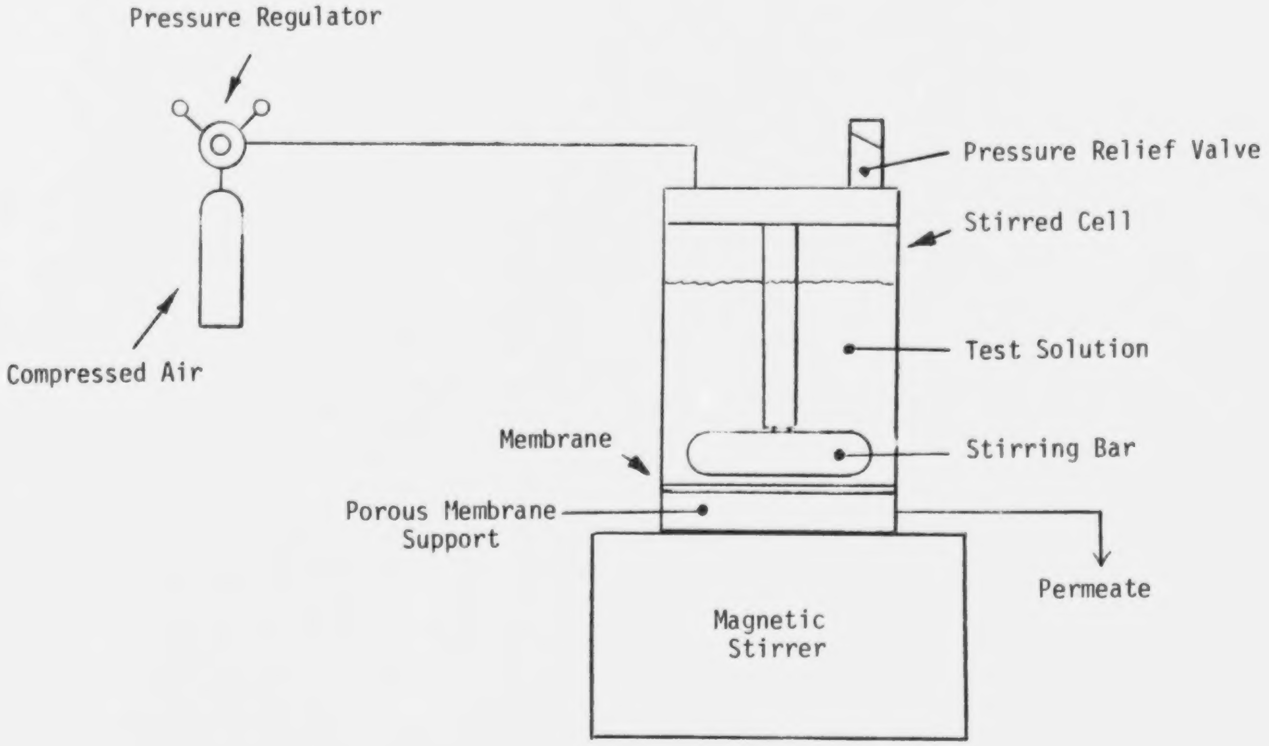

Pressure Regulator
Compressed Air
Pressure Relief Valve
Stirred Cell
Test Solution
Stirring Bar
Membrane
Porous Membrane Support
Permeate
Magnetic Stirrer

Figure 16. Detail of stirred-cell testing apparatus.

- Apparent color rejection: The apparent color rejection was obtained using equation 1:

$$R(\%) = \left(1 - \frac{C_p}{C_f}\right) \times 100 \qquad (1)$$

where

C_p = Concentration in the permeate

C_f = Concentration in the feed

- Intrinsic Rejection: Membrane rejection is an intrinsic property and may not be derived directly from test data under varying feed concentration conditions. Using certain assumptions (i.e. rejection is independent of concentration and osmotic pressure effects are insignificant) the intrinsic rejection can be predicted from the following equation which is derived in Appendix B:

$$R_a = 1 - \left[\frac{(1-Y_1)^{1-R_i} - (1-Y_2)^{1-R_i}}{Y_2 - Y_1} \right] \qquad (2)$$

where

R_a = Apparent rejection

R_i = Intrinsic rejection

Y_1 = Conversion at beginning of sample (50 ml permeated)

Y_2 = Conversion at end of sample (100 ml permeated)

Conversion is defined as follows:

$$Y = \frac{Vo - V}{Vo} \qquad (3)$$

where

Vo = initial volume in cell

V = volume in cell at time t

- Salt rejection: The salt rejection was obtained using equation (1) where

C_p = Conductivity of the permeate

C_f = Conductivity of the feed

MULTIPLE CELL TESTS

When several membrane types were being compared a multiple stirred cell test system was employed. This system is shown schematically in Figure 17. Caustic extraction filtrate was charged to the feed tank and pumped through 4 ultrafiltration stirred cells (Amicon Model 202), in series, by a piston-type positive displacement pump (Chemtrix Model 7800C). An accumulator dampened pressure fluctuations and a pressure gauge indicated the inlet pressure to the first test cell. A back pressure regulator maintained the operating pressure at 5.2 atm (75 psig). The permeate from the four cells and the concentrate from the fourth cell were all returned to the feed tank (i.e., total recycle operation). These tests were performed at ambient temperature.

MEMBRANE CASTING SOLUTION PREPARATION

The WRP casting solutions used to prepare flat-sheet membranes for laboratory evaluation were formulated by mixing 500 grams of the required chemicals in a one liter sample jar, sealing the jar, and placing the solution on a roller apparatus until a solution which appeared to be homogenous was obtained. The following chemicals were used in preparing the solutions:

Polymer:	Polysulfone (Union Carbide #P3500)
Solvent:	N-methyl-pyrrolidinone
Non-Solvent:	Tetrahydrothiophene-1,1-dioxide (Eastman #P9323)

Mixing times of 2-14 days were required to obtain solution homogeneity. Once the solution was homogenous its viscosity was measured and the membrane was cast. Casting was performed by drawing the solution over a sheet of Remay (which was taped to a glass plate) with a Gardner knife set to yield a solution thickness of 10 mils above the backing. The backing was then removed from the glass plate, held in a vertical position for 30 seconds and gelled in a room temperature bath containing either deionized water or a 0.01 weight % solution of dodecyl sulfate, sodium salt (Aldrich #75,192-2) in deionized water.

SAMPLING AND ANALYSIS

Samples were collected for two purposes during the program. First, samples of the three process streams of interest; hardwood and pine pulp washing decker effluents and pine bleachery caustic extraction filtrate, were analyzed at least daily to provide statistical data on composition and variations in composition on the various streams. Secondly, the operational streams were sampled during test module stand or pilot plant operation. These samples were taken of the feed stream, recycle stream,

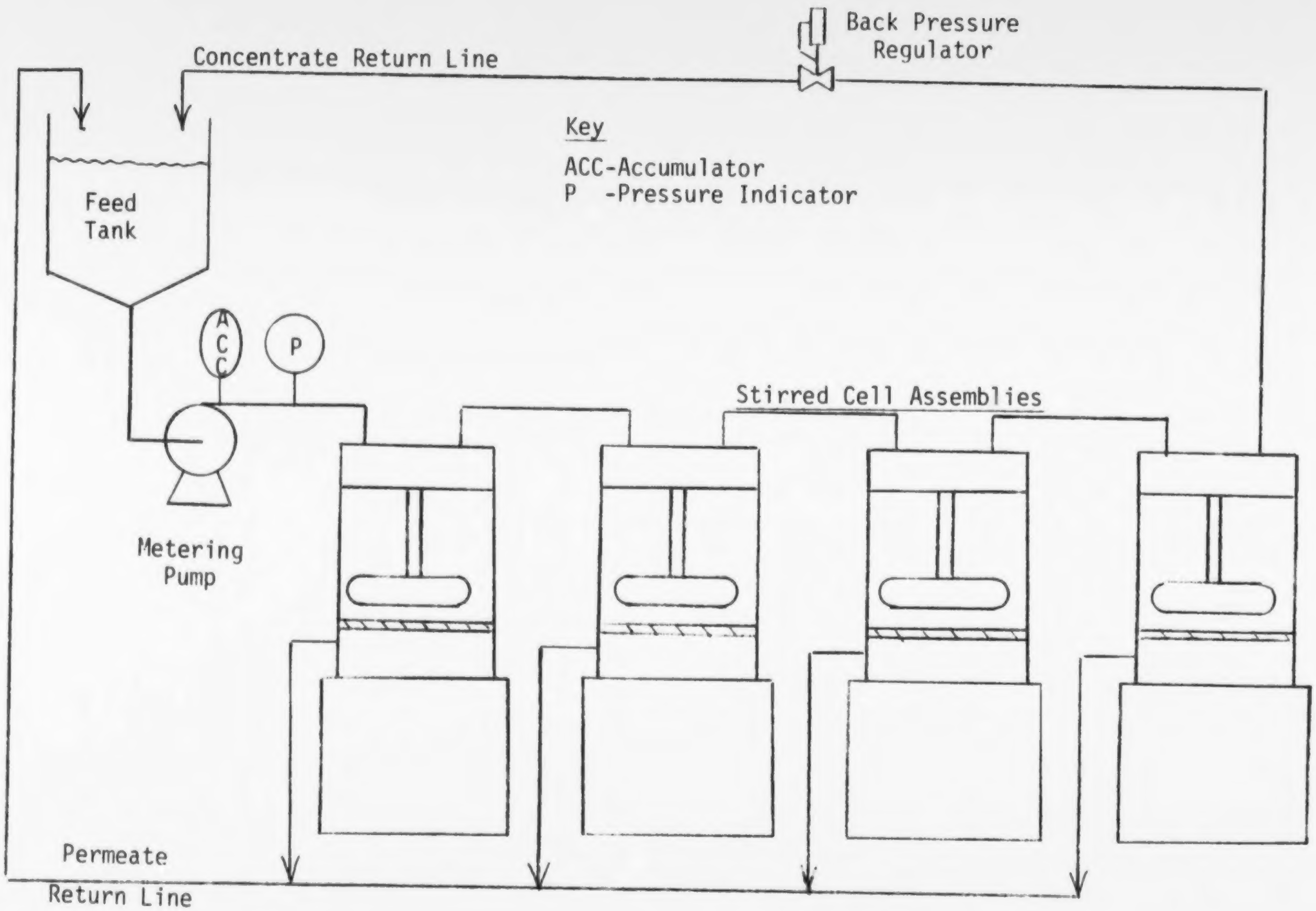

Back Pressure Regulator
Concentrate Return Line
Key
ACC-Accumulator
P -Pressure Indicator
Feed Tank
ACC
P
Stirred Cell Assemblies
Metering Pump
Permeate Return Line

Figure 17. Flow schematic for stirred cell total recycle tests.

concentrates (from each stage) and permeates (individual module and composites). All samples were taken in clean glass bottles rinsed several times with the stream to be analyzed after purging the sample lines.

The routine tests run on all samples were:

- temperature,
- pH,
- color,
- suspended solids, and
- total solids.

Standard methods for these analyses are listed in Table 1; brief descriptions follow.

Temperature: The temperature was measured with a mercury thermometer on the stream during sample taking.

pH: The pH was measured with a Beckman Zeromatic pH meter with standard glass and calomel electrodes.

Color: Sample had pH adjusted to 7.6, filtered through a 0.8 micron filter disc, and absorbance measured at 465 mμ on a Spectronic 20 spectrophotometer. Standard Pt/Co solution used for calibration.

Suspended solids: (Gravimetric) - measured volume of sample vacuum filtered through dried, preweighed 0.4 micron filter disc. Discs plus sample dried 4 hours at 105°C, cooled in a dessicator and reweighed.

Total solids: (Gravimetric) - Weighed sample dried at 105°C for 4 hours, cooled in a dissicator and reweighed.

Composite samples of the feed stream, the concentrates, and the permeates for 1.2X, 10X and 50X concentration during tubular membrane pilot operation were collected. These samples were submitted to Galbraith Laboratories, Inc. for the following analyses:

- Specific gravity
- Total solids
- Volatile solids
- Ash
- Calcium
- Iron
- Aluminum
- Sodium
- Sulfate
- Chlorine
- Ionic chloride

Standard methods employed for these assays are also given in Table 1.

TABLE 1. ASSAYS AND METHODS EMPLOYED DURING EXPERIMENTAL PROGRAM

Constituent	Assay Method	Reference
Aluminum	Atomic absorption	SM 301A*
Calcium	Atomic Absorption	SM 301A
Chlorine	Titration	SM 409E
Color	Colorimetric	TAPPI Method
Ionic chloride	Titration	Manufacturer's Manual
Iron	Atomic absorption	SM 301A
pH	Meter reading	SM 408C
Sodium	Flame photometric	SM 320A
Sulfate	Gravimetric	SM 427B
Suspended solids	Gravimetric	SM 208D
Total solids	Gravimetric	SM 208A
Volatile solids	Gravimetric	SM 208 E,G

* SM 30A (etc.) refers to procedure number in "Standard Methods for the Examination of Water and Wastewater," 14th Edition, APHA, 1975.

SECTION 6

RESULTS AND DISCUSSION

FEED CHARACTERISTICS AND PRETREATMENT

The characteristics of the feed stream and the effectiveness of pretreatment processes are important considerations in the operation of spiral-wound ultrafiltration modules. As such, the caustic extraction filtrate, and decker effluents were sampled on nearly a daily basis throughout a 21-month period (about 600 samples for each stream). Grab samples of the untreated streams were analyzed for:

-- pH;
-- Temperature;
-- Total solids;
-- Suspended solids; and,
-- Color.

Several pretreatment options were studied during this program, however, routine samples were collected of the Kisco depth filter effluent alone. These samples were only taken of the primary stream studied, caustic extraction filtrate. The pretreated stream samples were analyzed for suspended solids.

A summary of the mill effluent stream characteristics is presented in Table 2. A discussion of these overall summary data and average monthly data follows.

pH

The overall average pH of caustic extract, pine decker and hardwood decker effluents was 10.8, 10.9, and 9.9 units, respectively. Monthly average pH values are shown in Figure 18. The hardwood decker stream averaged a pH of 10 during nearly half of the sampling period. Excursion from this pH level were consistently on the low side until the final three months of the program. These latter readings are, however, closer to the 10.0 to 10.6 pH range observed for this stream in a prior investigation (32).

TABLE 2 . OVERALL SUMMARY OF FEED STREAM CHARACTERISTICS

Parameter	Caustic extraction filtrate range	Caustic extraction filtrate average	Pine decker effluent range	Pine decker effluent average	Hardwood decker effluent range	Hardwood decker effluent average
pH (units)	9.1-12.3	10.8	10.0-12.5	10.9	8.2-11.4	9.9
Temperature(°C)	29.4-52.2	40.8	30.0-65.0	48.3	30.0-61.7	43.9
Color (units)	4,670-29,300	18,450	666-13,000	4,740	2,370-34,000	13,600
Total solids (mg/l)	2,370-10,600	6,390	686-5,600	2,090	1,060-9,690	3,610
Suspended solids (mg/l)	10-253	62	13-427	67	40-660	251
Suspended solids following depth filter pretreatment (mg/l)	2-124	29	--	--	--	--

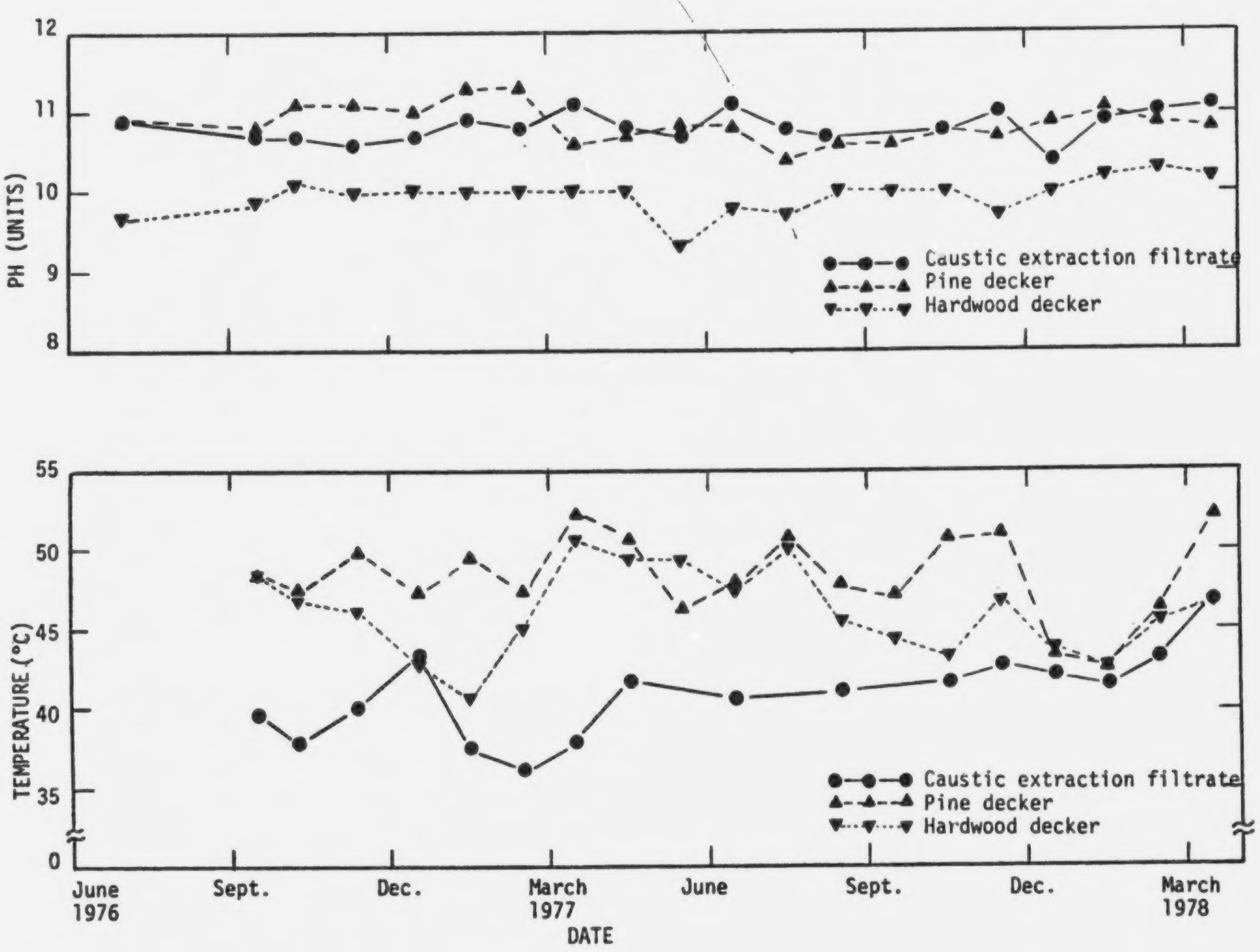


Figure 18. Monthly average temperature and pH levels of kraft pulp mill effluent streams.

The caustic extract and pine decker pH levels varied randomly during the nearly 2-year sampling period. The average values for the caustic extract stream were also below previously recorded levels. In 1972-73 the caustic extract stream pH ranged from 11.5 to 12 (32), as compared to a 1976-78 range of 9.1 to 12.3. The pine decker stream exhibited a wider pH range during the latter sampling but during both periods the average pH was about 10.9.

The wide fluctuations observed in the pH of these streams is probably due to recycling of other mill effluents back to the bleachery and decker operations. This recycling may also account for the lower pH levels in the caustic extract and hardwood decker streams.

Temperature

The monthly average temperatures of the streams of interest are also plotted in Figure 18. Cyclical fluctuations due to seasonal variations in temperature were not observed. Also, average temperature for all streams were below previous recorded values (48 to 57°C) (32). The overall average temperatures observed in this program were 40.8°C for caustic extraction filtrate, 48.3°C for pine decker effluent and 43.9°C for hardwood decker. The lower observed temperature for the caustic extraction filtrate may be due to the long uninsulated line (122 m) [400 ft] from source to test position.

Total Solids

Overall total solids contents averaged 2,090 mg/ℓ for the pine decker effluent, 3,610 mg/ℓ for the hardwood decker effluent and 6,390 mg/ℓ for the caustic extraction filtrate. Monthly average values are given in Figure 19. The pine decker effluent exhibited random variation in total solids loading and had a range of daily readings from 686 mg/ℓ to 5,600 mg/ℓ. The hardwood decker total solids content appeared to follow a seasonal pattern: low levels in winter, rising through spring then remaining at the average level through summer and into fall. However, the number of cycles observed was too few to verify this trend. The total solids range for this stream was 1,060 mg/ℓ to 9,690 mg/ℓ. The caustic extraction filtrate total solids level remained higher than average during the final six months of the sampling period. This suggests the expansion of water conservation measures at the mill bleachery. The total solids loading for the caustic extraction filtrate stream ranged from 2,370 mg/ℓ to 10,600 mg/ℓ.

Both the wide range of total solids readings and the average loadings observed for the three streams are comparable with prior data.

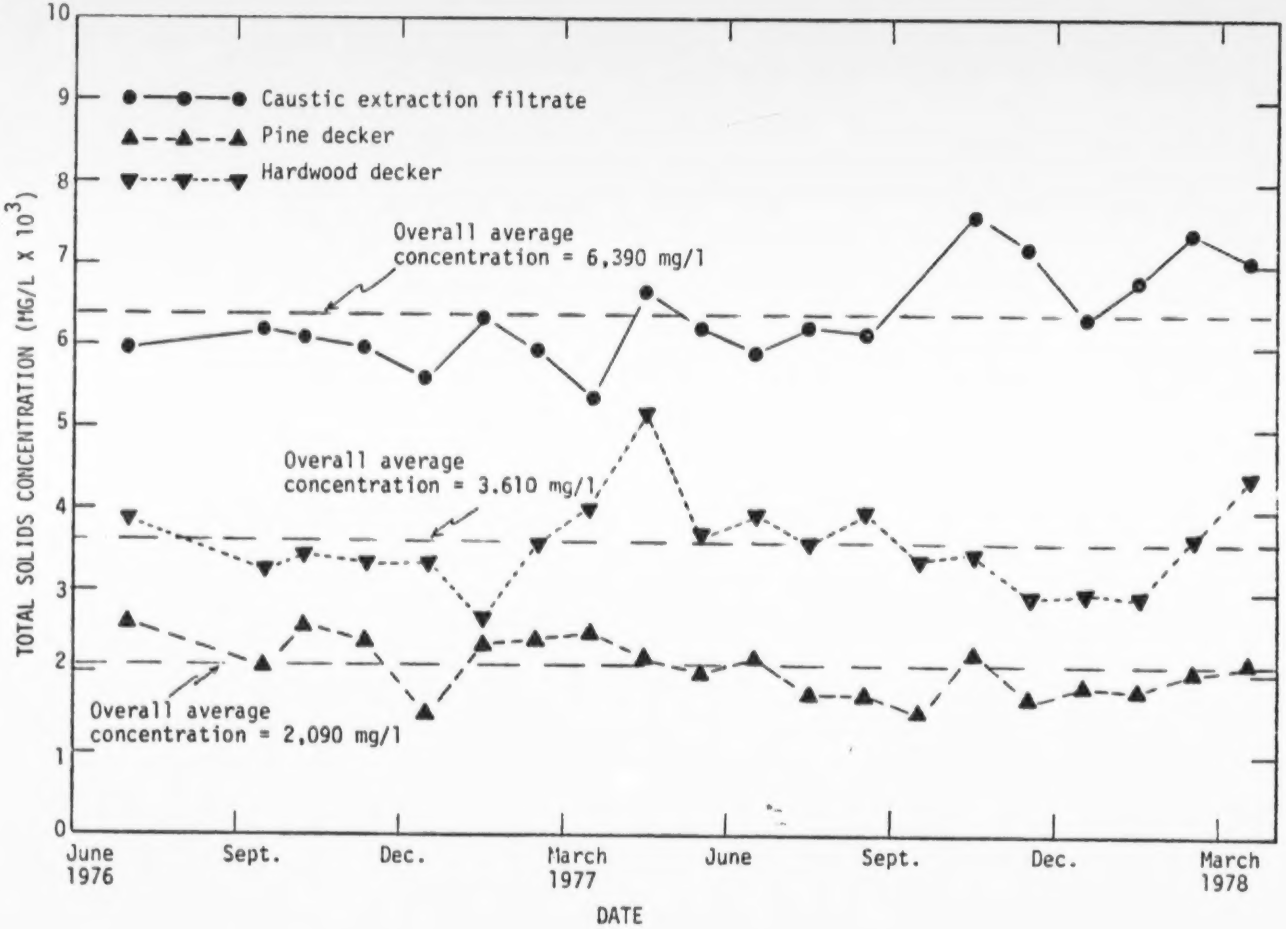


Figure 19. Monthly average total solids concentration for kraft pulp mill effluent streams.

Color

Plots of the monthly average color concentrations of the caustic extraction filtrate and decker effluents are shown in Figure 20. The pine decker streams averaged 4,740 color units and remained fairly stable on a monthly basis throughout the 21-month sampling program. The hardwood decker effluent varied widely in color concentration. Daily readings ranged from 2,370 to 34,000 color units. The pattern of monthly changes in color concentration for the hardwood decker closely parallels the pattern observed for the total solids content of this stream.

The stream with the highest color content is the caustic extraction filtrate. This stream averaged 18,500 color units with a range of 4,670 to 29,300 color units. The color content remained above average for the final six months of the sampling period, again signalling increased water conservation measures.

During the 1972-73 sampling program the average color contents of these streams were 28,000, 6,000 and 11,000 color units for the caustic extraction filtrate, pine decker and hardwood decker, respectively. For the caustic extract stream the current average color loading represents a 50% decrease from the past level. The current pine decker color concentration is about 25% lower than the previously measured level. Conversely, the hardwood decker color loading has increased by almost 20%. All of these comparisons point out the continually changing nature of the mill effluent streams.

Suspended Solids

Plots are shown in Figure 21 of the monthly average suspended solids concentrations in the three streams. An average loading of 62 mg/ℓ was recorded for the caustic extraction filtrate. This stream ranged on a daily basis from 10 to 253 mg/ℓ suspended solids. The pine decker effluent had a similar average, 67 mg/ℓ, and a somewhat wider range: 13 to 427 mg/ℓ. Monthly fluctuations in the suspended solids content for these streams were relatively minor.

The stream with the highest suspended solids loading and hence, potentially most troublesome for spiral-wound module ultrafiltration is the hardwood decker effluent. The spread in suspended solids readings for this stream was 40 to 660 mg/ℓ with an average level of 251 mg/ℓ. The variations in suspended solids loading for the hardwood decker do not correlate over the entire sampling period with the total solids and color content variations discussed above.

Caustic Extraction Filtrate Pretreatment

The effectiveness of depth filtration in removing suspended solids from caustic extraction filtrate was monitored throughout this program. Samples of the caustic extract stream were analyzed for suspended solids before and

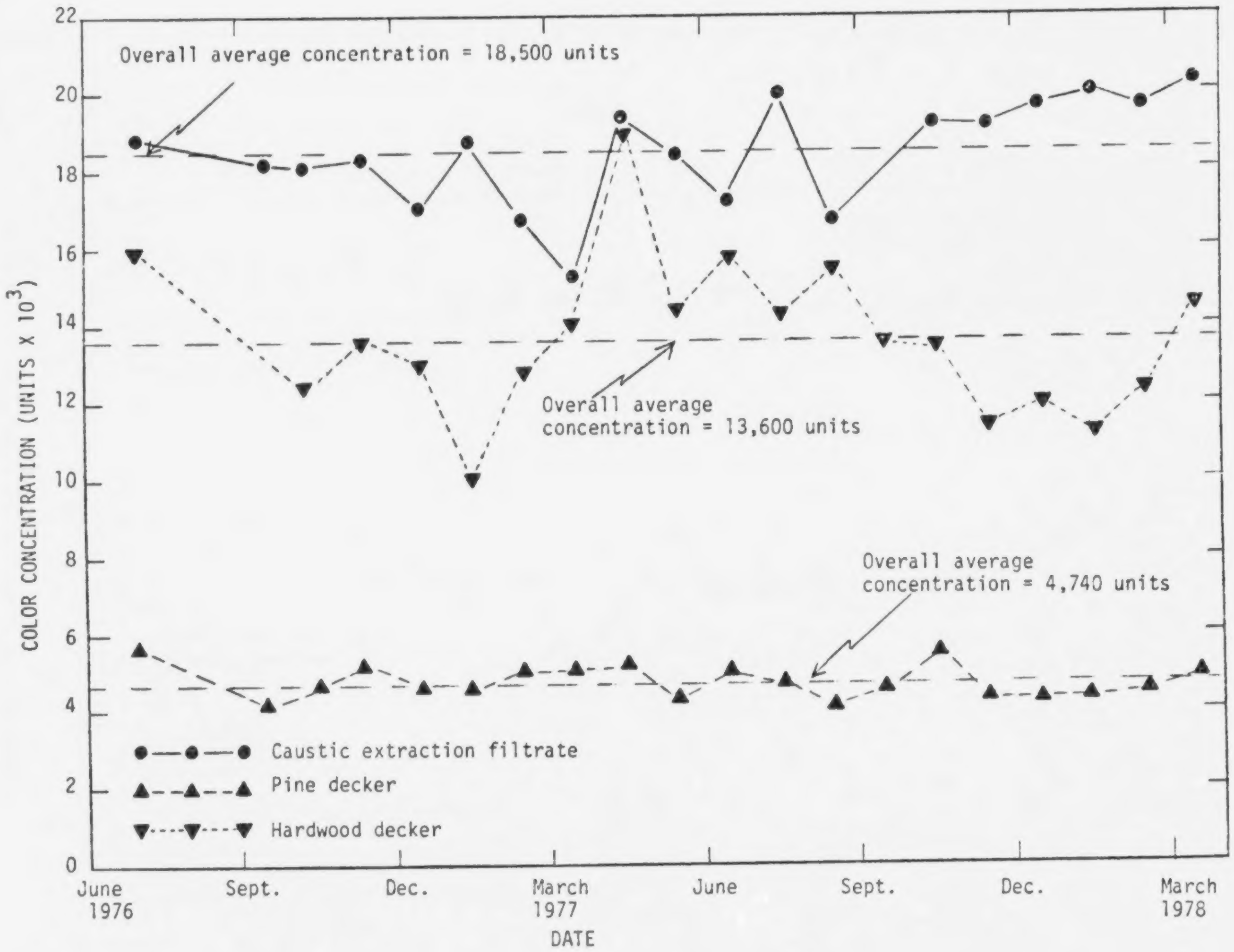


Figure 20. Monthly average color concentration of kraft pulp mill effluent streams.

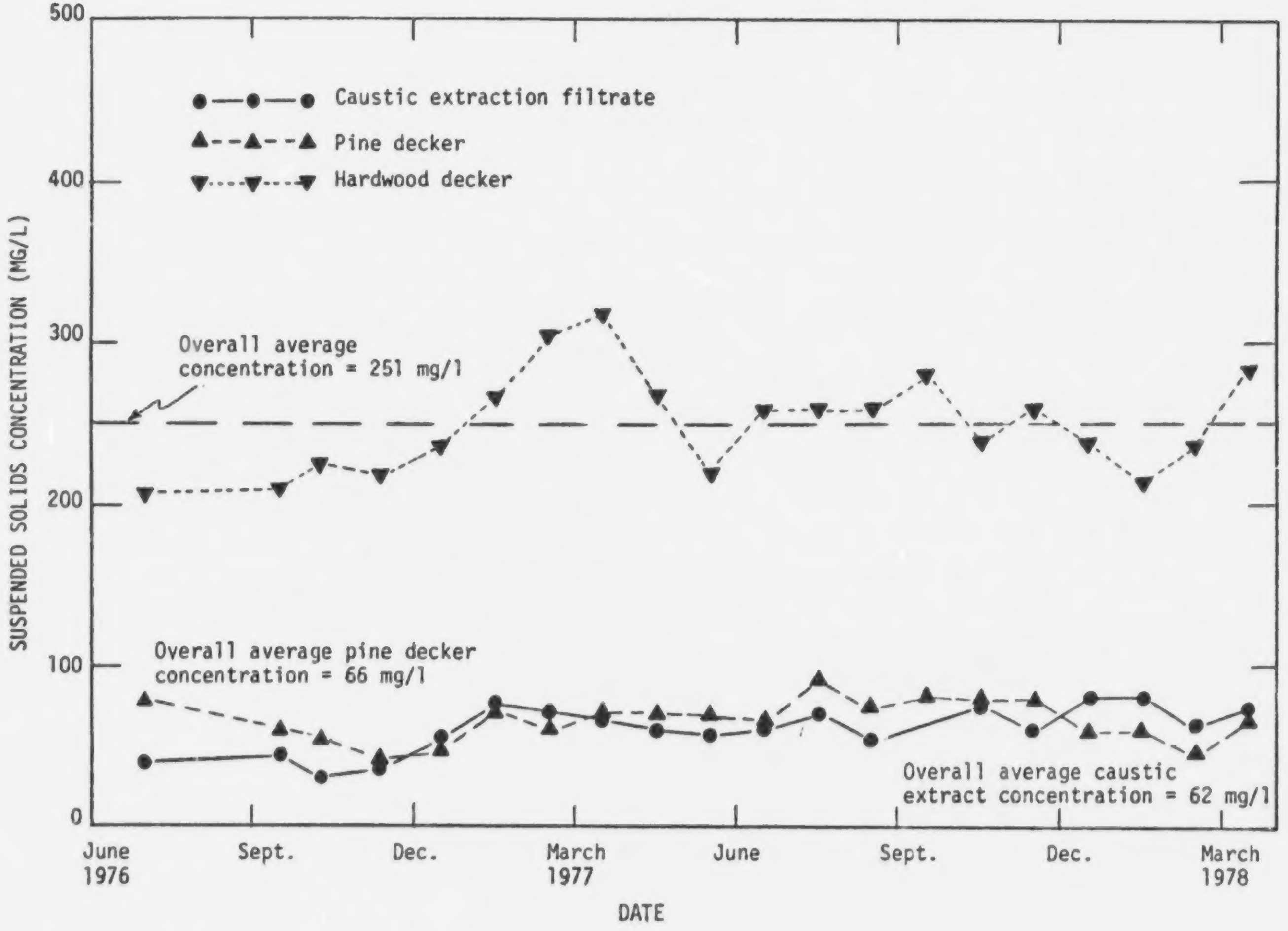


Figure 21. Monthly average suspended solids concentration of kraft pulp mill effluent streams.

after treatment by the Kisco depth filter. The results of these analyses are plotted in Figure 22 along with the calculated suspended solids removal efficiency for the filter. The average feed suspended solids loading was 60 mg/ℓ; the average filter effluent suspended solids loading was 29 mg/ℓ. An average removal efficiency of about 50% was achieved. This average removal efficiency is somewhat deflated due to periodic breakthrough in the filter column. Disregarding the April 1977 and February 1978 average figures (see Figure 22) the depth filter removal efficiency for suspended solids had a mean value of 57%.

Summary

In general, solids content, color, pH and temperature varied randomly from month-to-month. Wide fluctuations were observed on a daily basis. A seasonal pattern to effluent temperature changes was not observed. A cyclic pattern to the hardwood decker total solids and color concentrations began to take shape but would require further study to verify.

In terms of total solids and color the caustic extraction filtrate stream has the highest loadings. The suspended solids content of the hardwood decker effluent is about four times as high (251 mg/ℓ) as the pine decker (67 mg/ℓ) or caustic extraction filtrate (62 mg/ℓ) streams.

Due to equipment changes, process changes or water conservation measures the nature of the three effluents has changed over the past four to five years. These types of changes can be expected to occur in the future but should pose little, if any, problems for an ultrafiltration treatment system.

SELECTION OF PREFERRED MEMBRANE FOR COLOR REMOVAL

Introduction

The initial phase of this program involved selection of the preferred non-cellulosic membrane for color removal from kraft pulp mill effluents. The preferred membrane was selected from laboratory tests with the following candidate systems:

- Commercially available UF membranes coated with cross-linking agents to improve rejection characteristics;
- Non-commercial polysulfone-based membranes coated with cross-linking agents to improve rejection characteristics;
- Non-commercial polysulfone-based membranes tailored specifically to color removal applications (non-coated); and
- Non-commercial interpolymer fixed-charge membranes.

All candidate membranes were evaluated in flat-sheet form in stirred-cell tests (Screening Tests). Several of the coated commercially available membranes were further evaluated in the tubular geometry (Parametric Tests).

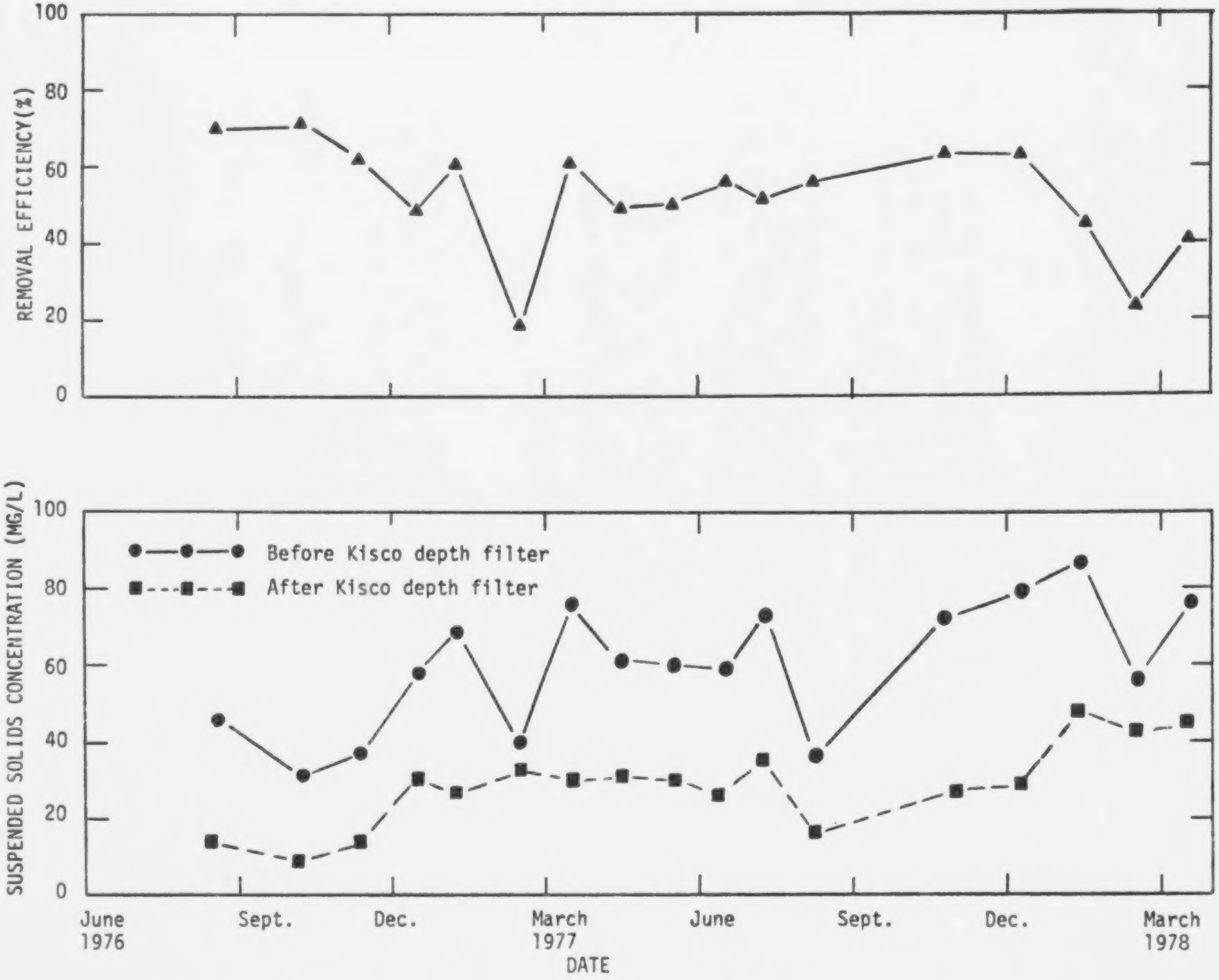


Figure 22. Caustic extraction filtrate prefiltration data (monthly averages).

Following selection of the preferred membrane, a full-scale spiral-wound module was fabricated and laboratory tested before module preparation for on-site piloting.

All membrane selection tests were performed with caustic extraction filtrate shipped from the Canton Mill. This stream contains lower molecular weight color bodies than the decker effluents and, as such, is a "worst case" feed stream.

Membrane Screening Tests

The first series of membrane screening tests were performed with Abcor, Inc. types HFD, HFM and WRP membranes. The type HFD and HFM membranes are commercially available, non-cellulosic ultrafiltration membranes. WRP membrane is a research polysulfone-based material. Interpolymer fixed-charge membranes (provided by H. Gregor, Columbia University, New York) were not available for the initial screening tests.

Thirty-five different membrane-coating combinations were prepared from the basic HFD, HFM and WRP membranes. The coating formulations were based on proprietary Abcor technology with chemical cross-linking agents. The listing of the membranes prepared and their stirred-cell performance characteristics are given in Table 3. As pointed out below coated membranes failed to maintain their performance characteristics and were not selected for pilot testing.

General trends can be observed from these screening tests (see Table 3). First, WRP membrane rejection of color bodies exceeds that of HFD and HFM membranes. No HFM membrane/coating combination provided intrinsic color rejection as high as 99%. The best rejecting HFM membrane (HFM-GH 500) exhibited 98.5% rejection with an associated average flux of 0.86 m^3/m^2-day (5.1 atm) [21 gfd (75 psig)]. Two HFD membranes reached or exceeded 99% intrinsic color rejection (HFD-FH 500 and HFD-GH 500) however these membranes had flux levels of 0.26 m^3/m^2-day (6.3 gfd) and 0.53 m^3/m^2-day (13 gfd), respectively. Nine of the 13 WRP membranes had intrinsic rejections exceeding 99% with several membranes showing 99.5% rejection or better. Flux levels for these WRP membranes were >0.62 m^3/m^2-day (15 gfd) in 6 cases, and as high as 1.97 m^3/m^2-day (48 gfd).

Clearly, the most important characteristics in determining membrane performance are rejection and capacity (flux). The preferred membrane would have a high rejection and a high capacity. However, in actual practice it is found that high rejection membranes have relatively low fluxes. This is shown graphically in Figure 23 in which the intrinsic color rejection is plotted versus membrane flux. Obviously a trade-off exists between rejection and flux. The data in Figure 23 indicate that the coated WRP membranes achieve significantly higher flux levels at the same degree of color removal than either the coated HFD or the coated HFM membranes. This

TABLE 3 . RESULTS OF STIRRED CELL SCREENING TESTS

Membrane	Average flux m^3/m^2-day (gfd)	Apparent color rejection, %	Intrinsic color rejection, %*	Salt† rejection,%
HFD	3.36(82)	84.0	90.3	0
HFD-FN250	3.89(95)	82.2	89.2	0
HFD-FN500	1.60(39)	89.0	93.5	12
HFD-GN250	3.44(84)	90.3	94.3	3
HFD-GN500	2.46(60)	92.2	95.5	12
HFD-FH50	1.52(37)	96.8	98.2	11
HFD-FH250	1.27(31)	98.1	98.9	35
HFD-FH500	0.25(6.3)	98.3	99.1	42
HFD-GH50	1.80(44)	96.0	97.7	24
HFD-GH250	1.23(30)	97.0	98.3	6
HFD-GH500	0.53(13)	98.2	99.0	32
HFM	4.26(104)	78.0	86.4	0
HFM-FN250	3.81(93)	84.3	90.6	0
HFM-FN500	2.91(71)	88.0	92.8	12
HFM-GN250	3.49(85)	87.8	92.7	12
HFM-GN500	2.34(57)	92.0	95.4	24
HFM-FH50	1.56(38)	96.6	98.4	22
HFM-FH250	2.01(49)	93.9	96.5	12
HFM-FH500	1.39(34)	94.8	97.0	12
HFM-GH50	3.73(91)	89.1	93.6	12
HFM-GH250	1.76(43)	95.4	98.0	5
HFM-GH500	0.86(21)	97.3	98.5	41
WRP	1.60(39)	94.4	96.8	14
WRP-FN50	1.60(39)	95.6	97.5	41
WRP-FN250	1.60(39)	98.3	99.0	47
WRP-FN500	1.48(36)	97.6	98.6	32
WRP-GN50	1.07(26)	97.9	98.8	33
WRP-GN250	1.97(48)	98.4	99.1	35
WRP-GN500	1.07(26)	98.7	99.3	47
WRP-FH50	0.62(15)	98.5	99.2	62
WRP-FH250	0.38(9.3)	99.3	99.6	70
WRP-FH500	0.36(8.7)	99.5	99.7	45
WRP-GH50	0.98(24)	99.1	99.5	30
WRP-GH250	0.78(19)	99.1	99.5	47
WRP-GH500	0.17(4.1)	99.1	99.5	50

* Calculated

† Using $MgSO_4$

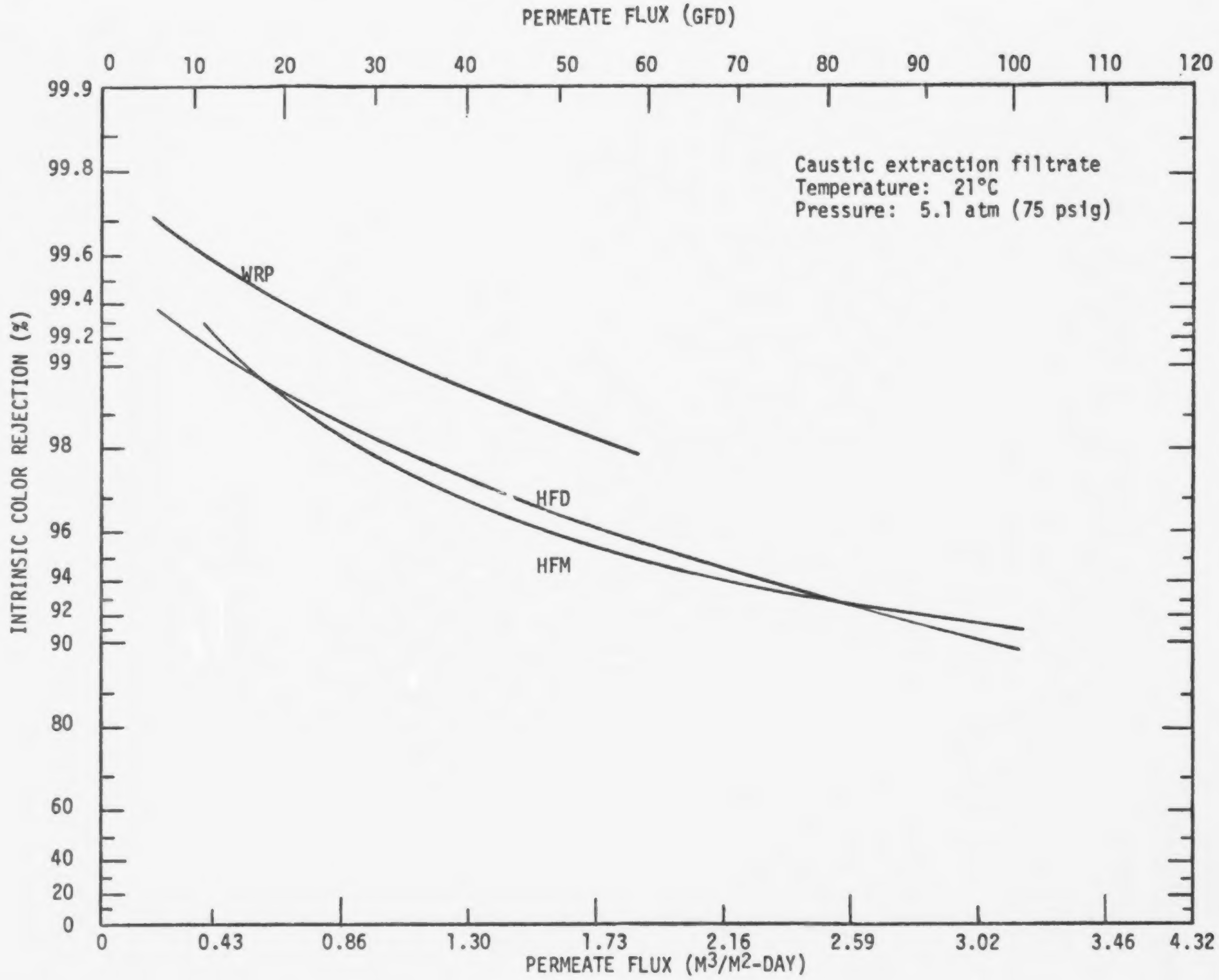


Figure 23. Intrinsic color rejection versus flux during stirred cell tests.

suggests that the WRP membrane is probably the preferred substrate for the treatment of caustic extract effluents; however, parametric studies were performed with selected HFD and HFM membranes to verify this finding.

Parametric Tests

The purpose of the parametric tests was to determine the effects of temperature, pressure and feed flow rate on the performance of HFD and HFM membranes with different coatings. Three HFM and two HFD membrane/coating combinations were tested in 25.4 mm (1 in) diameter x 1.52 m (5 ft) long tubular assemblies. Only one of each type of treated HFD and HFM tubular membrane was made. WRP tubular membranes were not available for testing at this time. A listing of the parametric tests conducted is given in Table 4.

Flux data for the coated HFD membranes throughout tests 1 to 6 are shown as a function of cumulative operating time in Figure 24. A general trend of increasing membrane flux with time can be observed. Also, within each total recycle test a flux increase with time typically occurred. Looking specifically at the data from tests 4 and 6: a 3-fold increase in HFD - FH 250 flux and a 2-fold increase in HFD - GH 500 flux took place between these two tests. Both tests were conducted at 60°C (140°F) and 3.4 atm (50 psig) inlet pressure. The feed flow rate was 163.5 m^3/day (30 gpm) for test 4 and 109 m^3/day (20 gpm) for test 6. On the basis of the lower feed flow rate a reduction in membrane flux would have been expected in test 6 as opposed to test 4. Since the opposite effect occurred, and because of the generally increasing flux performance the final three parametric tests were conducted at the same conditions as test 1 (49°C, 3.4 atm, 163.5 m^3/day) [120°F, 50 psig, 30 gpm].

Both HFD membrane flux and membrane color rejection are plotted as functions of cumulative operating time in Figure 25 for tests 1, 7, 8 and 9. Flux increased with time while color rejection decreased. Similar trends were observed for the coated HFM membrane series (see Appendix C). These results indicated that the membrane coatings were degrading (possibly being physically removed) as a function of operating time. In view of these results and the excellent data obtained with the WRP membrane series in the initial screening tests further evaluation of coated HFD and HFM membranes was deferred.

Optimization of the WRP Membrane Series

Introduction--

Two conclusions were evident from the initial screening tests and the parametric studies: The WRP membrane was preferred over the HFD and HFM membranes and the membrane coating formulations were subject to degradation with time. The next step in selecting the preferred membrane for color removal from kraft pulp mill effluents was to determine the preferred uncoated WRP membrane formulation.

TABLE 4 . PARAMETRIC STUDIES TEST MATRIX (25.4 MM DIAMETER X 1.52 M LONG TUBULAR MEMBRANES)

Test	Operating temperature, °C (°F)	Operating pressure, atm (psig)	Feed flow rate, m^3/day (gpm)	Test duration hours
1	49 (120)	3.4 (50)	163.5 (30)	24
2	49 (120)	5.1 (75)	109 (20)	25
3	60 (140)	5.1 (75)	109 (20)	32
4	60 (140)	3.4 (50)	163.5 (30)	20
5	60 (140)	5.1 (75)	163.5 (30)	19
6	60 (140)	3.4 (50)	109 (20)	22
7	49 (120)	3.4 (50)	163.5 (30)	24
8	49 (120)	3.4 (50)	163.5 (30)	115
9	49 (120)	3.4 (50)	163.5 (30)	21

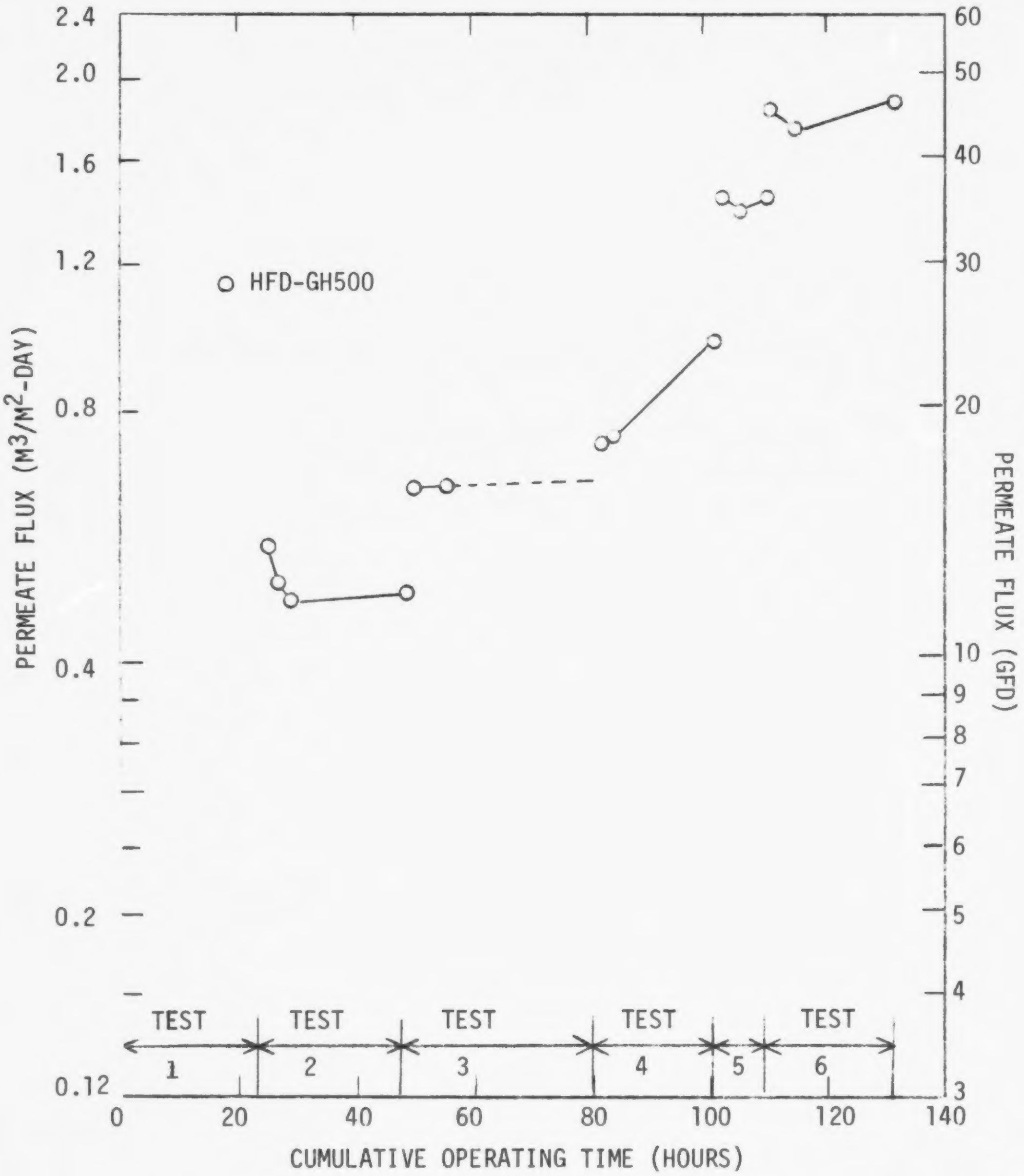


Figure 24 . Coated HFD membrane flux data obtained during parametric studies.

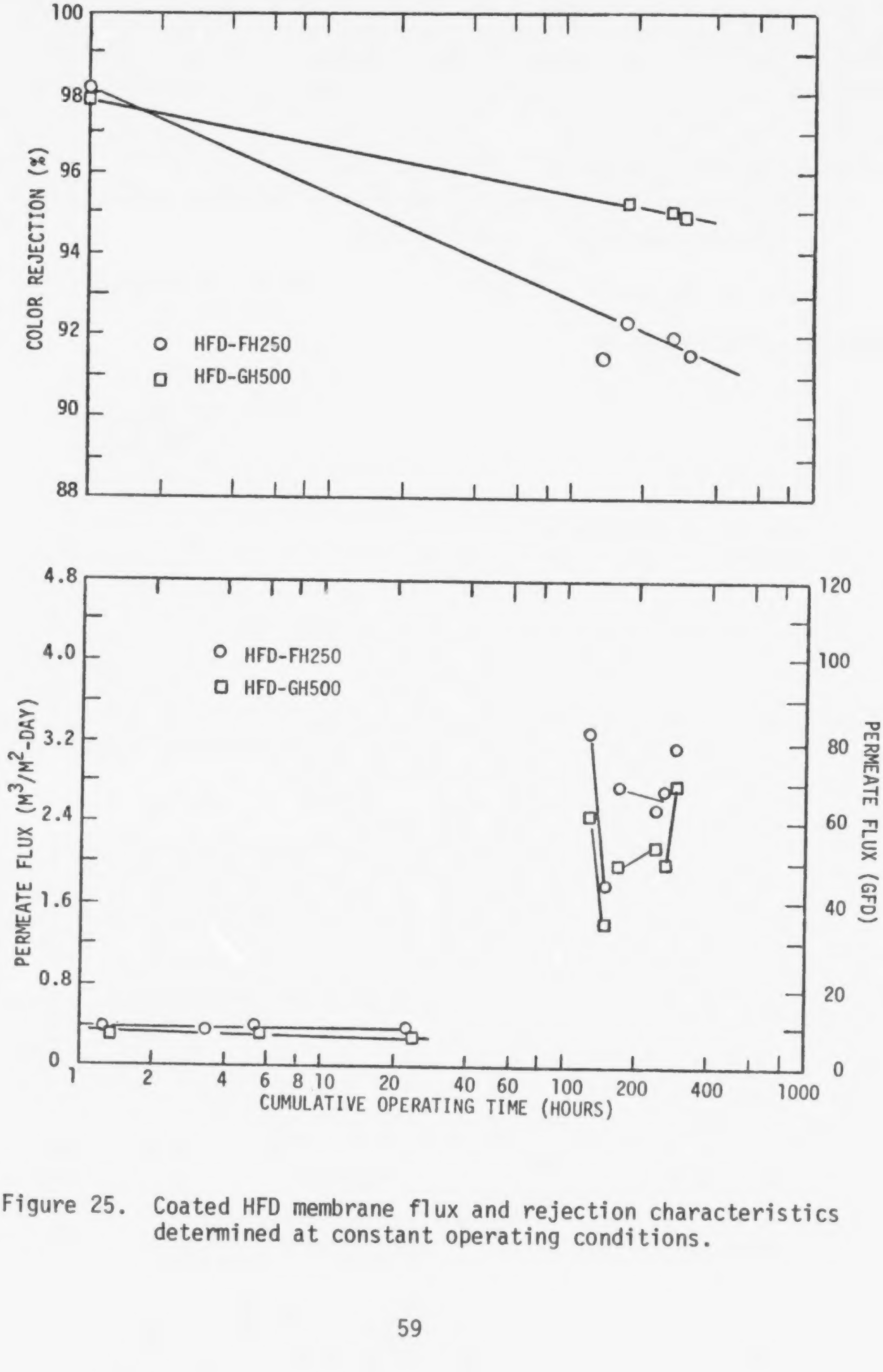


Figure 25. Coated HFD membrane flux and rejection characteristics determined at constant operating conditions.

WRP membranes can be formed using different casting solutions containing the same base polymer, polysulfone. Differences in the casting solutions (and casting conditions) result in the formulation of membranes with different structures and hence different performance characteristics. The variables examined in tailoring the WRP membrane to mill effluent treatment were:

- polymer concentration in the casting solution;
- non-solvent concentration in the casting solution; and,
- surfactant presence in gelation bath.

The following method of labelling the membranes was derived:

Membrane designation WRP __ _ __

$$100\left(\frac{\text{Weight of non-solvent in casting solution}}{\text{Weight of non-solvent + solvent in the casting solution}}\right)$$

W - gelled in a surfactant solution
N - gelled in deionized water

30-100X (Weight fraction of polysulfone in the casting solution)

As an example WRP10W33 was formed using a casting solution containing 20 weight % polysulfone 26.7 weight % non-solvent, and 53.3 weight % solvent that was gelled in a bath containing surfactant.

Results and Discussion--

A summary of the membrane performance characteristics is given in Table 5. The data shown for each membrane type are based on test results (5.1 atm, 21°C) with four membranes. An explanation of the calculation methods employed in determining the numbers presented is as follows:

1. Water flux: The four values obtained were averaged.
2. Process Flux at 3 Hours: The data from each of the four tests were plotted on semilog paper (linear flux; log time) and a least squares best fit was performed on each set of data. The flux level for each membrane at three hours was read from the graph and the resulting numbers were averaged.
3. Magnesium Sulfate Rejection: The four values obtained were averaged.
4. Color Rejection: The highest and lowest rejection values obtained were dropped and the remaining rejection values were averaged.

TABLE 5. PERFORMANCE OF WRP MEMBRANES (AT 5.1 ATM, 21°C)

Membrane	Water flux, m^3/m^2-day (gfd)	Process flux @ 3 hours, m^3/m^2-day (gfd)	Magnesium sulfate rejection,%	Color rejection,%
WRP 00 N 00	0.07 (1.6)	0.03 (0.8)	--	94.2
WRP 00 W 00	0.09 (2.1)	0.05 (1.2)	18	82.8
WRP 05 N 00	0.68 (16.5)	0.38 (9.3)	32	96.6
WRP 05 W 00	0.71 (17.4)	0.33 (8.0)	26	96.5
WRP 06 N 33	2.39 (58.4)	0.70 (17.0)	32	93.3
WRP 06 W 33	1.80 (43.9)	0.57 (13.8)	26	96.8
WRP 08 N 33	2.72 (66.4)	0.62 (15.0)	33	96.6
WRP 08 W 33	3.05 (74.4)	0.63 (15.4)	25	97.8
WRP 10 N 00	3.77 (92.0)	0.76 (18.6)	31	97.1
WRP 10 N 33	3.18 (77.5)	0.67 (16.4)	26	97.2
WRP 10 W 00	3.63 (88.6)	0.78 (19.1)	34	96.3
WRP 10 W 33	3.90 (95.2)	0.70 (17.0)	32	97.6
WRP 11 N 33	4.26 (104)	0.85 (20.8)	30	96.5
WRP 11 W 33	3.44 (83.8)	0.80 (19.4)	34	97.0
WRP 11 W 50	4.35 (106)	0.93 (22.6)	34	97.0
WRP 14 W 50	5.95 (145)	0.98 (23.9)	26	92.7
WRP 17 W 67	5.21 (127)	1.00 (24.4)	35	91.8

The best results were obtained with the WRP08W33 and the WRP11W33 membranes. Membrane WRP08W33 yielded a color rejection of 97.8% and a process flux after three hours of 0.63 m^3/m^2-day (15.4 gfd) when processing caustic extraction filtrate. Membrane WRP10W33 yielded a color rejection of 97.6% and a process flux after three hours of 0.7 m^3/m^2-day (17.0 gfd).

The effect of polymer concentration on water flux and process flux is shown in Figures 26 and 27. As expected, flux declined with increasing polymer concentration. A linear relationship is observed in both cases. Little or no effect on membrane flux is evident from the presence of non-solvent in the casting solution or from surfactant in the gelation bath.

The effect of all variables examined on membrane color rejection is given in Figure 28. Rejection with respect to polymer concentration went through a maximum in all cases in the range of 20% to 24% by weight polymer. The best rejections were obtained with membranes cast from solutions containing non-solvent and gelled in a bath containing surfactant. It should be noted that most of the membranes cast with non-solvent in the casting solution had a solvent to non-solvent ratio of 2:1 (i.e., the effect of non-solvent was not thoroughly investigated).

It can be concluded from these data that high rejection-moderate flux ultrafiltration membranes can readily be made from polysulfone based casting solutions. Color removals from caustic extraction filtrate in excess of 97.5% at flux levels of 0.7 m^3/m^2-day (17 gfd) were attained at 5.1 atm (75 psig). These results can be compared to 90.3% color removal at 1.6 m^3/m^2-day (39 gfd) for one HFD flat sheet membrane and 86.4% color removal at 2.0 m^3/m^2-day (49 gfd) for the HFM membrane.

The preferred WRP membrane was determined to be WRP09W33. This membrane was not cast during these tests but was selected based on the data plots of Figures 26, 27 and 28. Theoretically this membrane would have higher flux than WRP08W33 (>0.63 m^3/m^2-day) [15.4 gfd] and higher rejection than WRP10W33 (>97.6%). WRP09W33 would be gelled in a bath containing 0.01 weight % dodecyl sulfate, sodium salt and be cast on a Remay backing using the following solution:

Solvent:	N-methyl-pyrrolidinone	52.7 grams
Non-Solvent:	Tetrahydrothiophene-1,1-dioxide	26.3 grams
Polymer:	Polysulfone	21.0 grams

Interpolymer Fixed Charge Membranes

Introduction--

Interpolymer fixed charged membranes were prepared by H. Gregor (Columbia University, New York) and tested at both Columbia and Abcor. All of these membranes were made in accordance with U.S. Patent 3,808,305 and

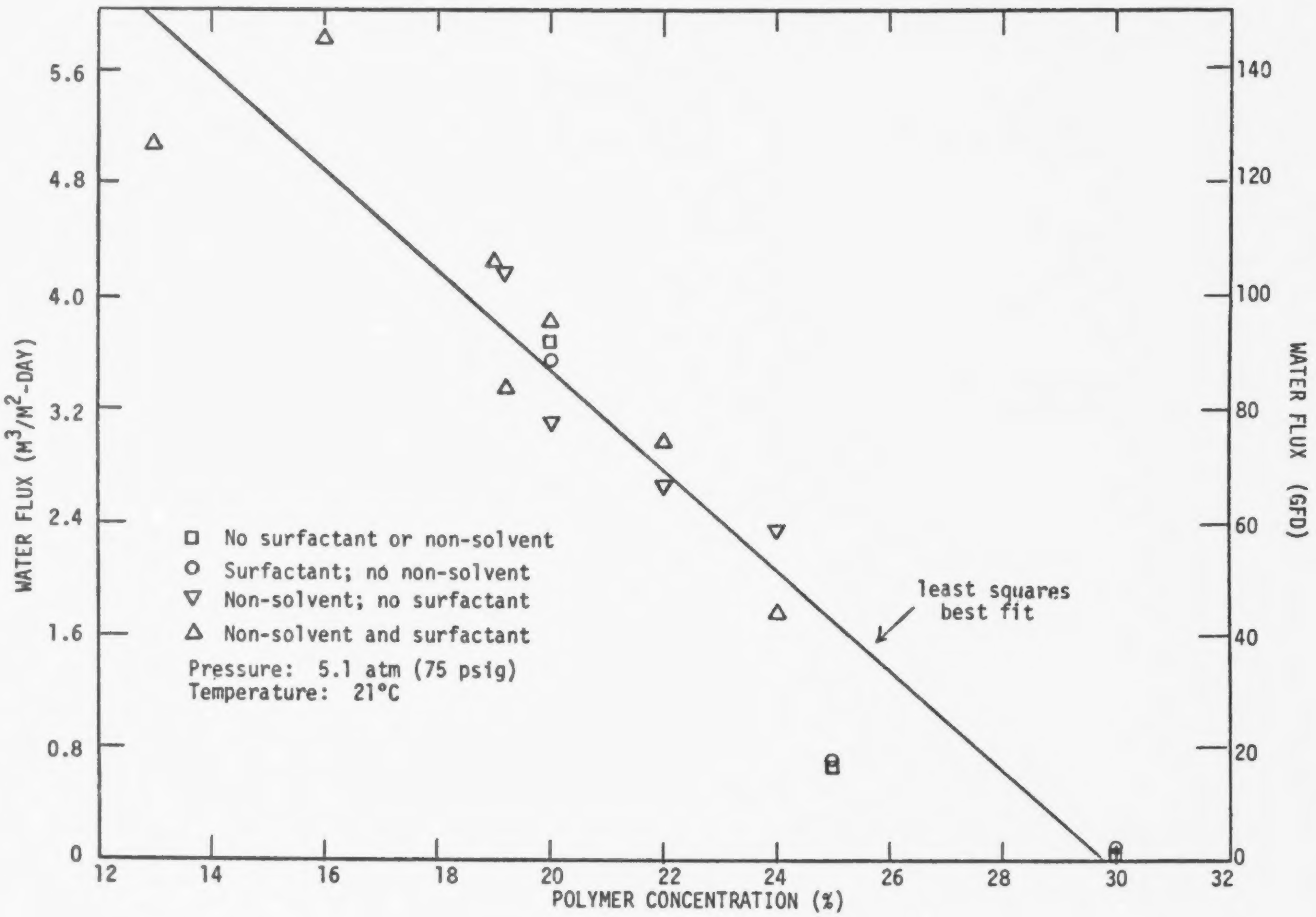


Figure 26. The effect of polymer concentration on WRP membrane water flux.

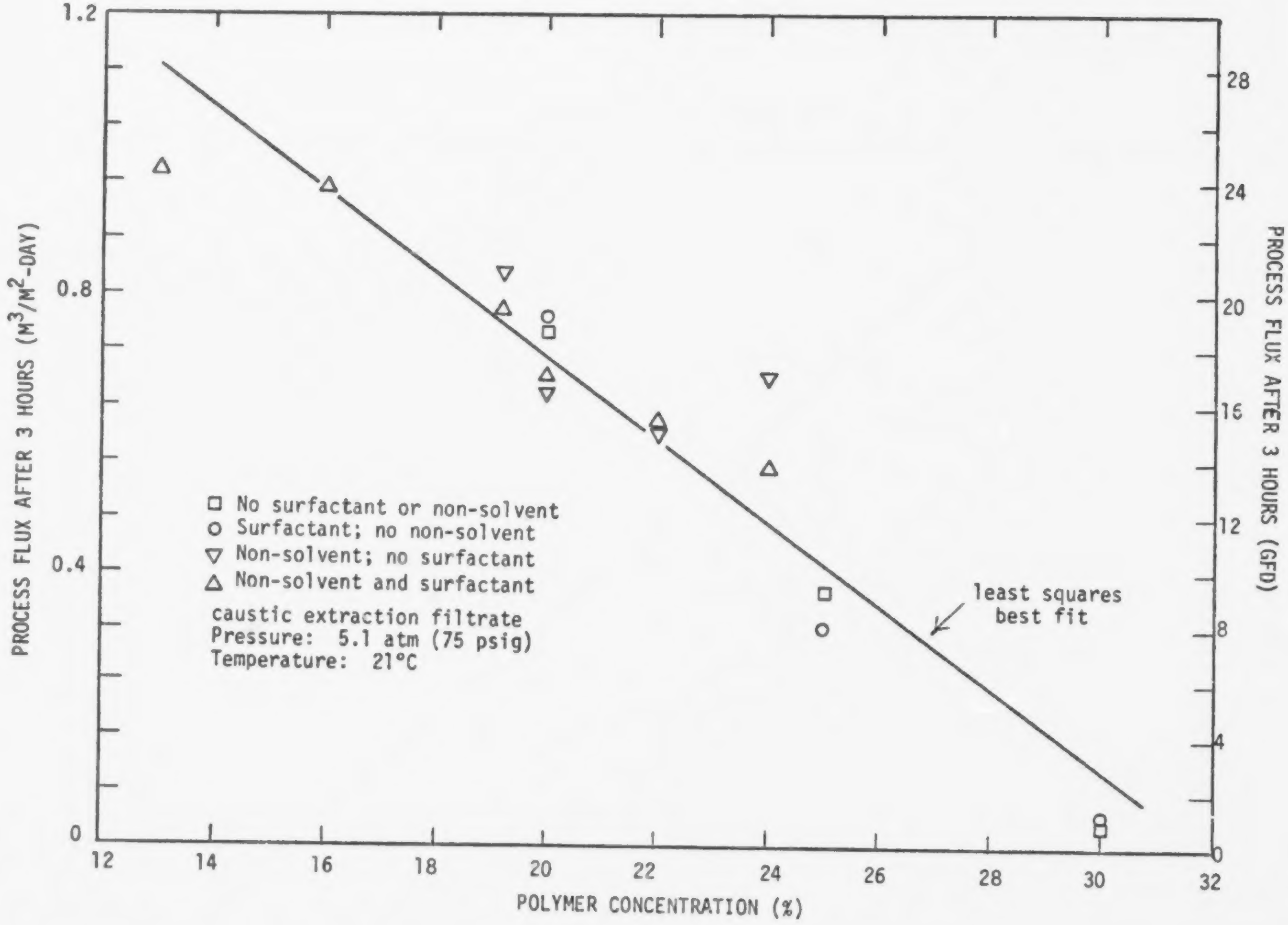


Figure 27. The effect of polymer concentration on WRP membrane process flux.

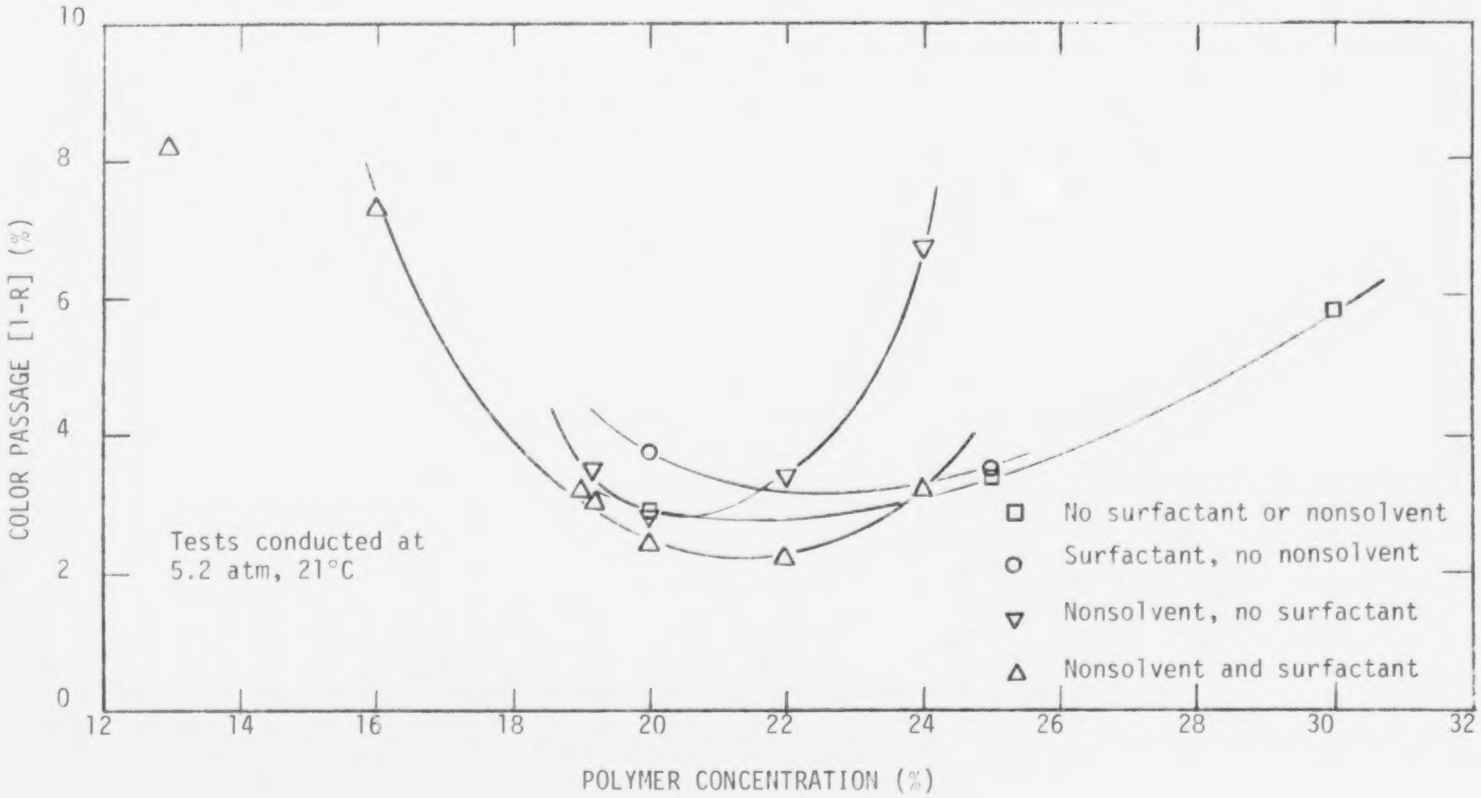


Figure 28. The effect of polymer concentration on WRP membrane color rejection.

consisted primarily of sulfonic acid polymers. Seven flat sheet (7.6 cm x 7.6 cm) membranes were tested at Abcor. The results of these studies are presented below. Tests performed by Dr. Gregor are documented in his Final Report, "Use of Sulfonic Acid Membranes for Treatment of Pulp and Paper Waste Streams". This report is presented, in its entirety, in Appendix D.

Short Term Tests--

A summary of the short term test results obtained with the interpolymer fixed charge membranes is given in Table 6. All membranes had only letter designations (e.g., A, B ... G), as received. Details on the membranes of most interest (A through E) can be found in Dr. Gregor's report.

In these 3-hour tests with caustic extraction filtrate the interpolymer fixed charge membranes displayed color rejection and process flux characteristics similar to the WRP membrane series. Process flux after 3 hours ranged from 0.42 to 0.71 m^3/m^2-day (10.2 to 17.4 gfd). Color rejection varied from 90.7% to 97.8%. Best results were obtained with membrane B which exhibited a process flux of 0.7 m^3/m^2-day (17 gfd) and a color rejection of 97.8%.

Long Term Tests--

The interpolymer fixed charge membranes were predicted (by their developer) to be non-fouling in nature. Since short term tests showed no practical differences between the WRP and the interpolymer fixed charge membranes, long term tests were conducted to determine the flux decline characteristics of each membrane type. The membranes selected for testing were those which had demonstrated superior performance in previous tests. These were interpolymer fixed charge membranes A and B, WRP10W33 and WRP11W33.

The flux decline curves for the polysulfone-based and sulfonic acid-based membranes are shown in Figure 29 for the 65 hour comparison test. Membrane flux decline rates were essentially identical even though the WRP series membranes showed intrinsically higher flux characteristics. The difference (i.e., increase) in WRP flux over previous trials is attributed to variation in the feed solution. In this test the WRP and the interpolymer fixed charge membranes were evaluated simultaneously giving a true comparison of both intrinsic membrane flux and rejection properties and flux decline characteristics.

Membrane color rejections during this test were:

Membrane A -	93.4%
Membrane B -	96.7%
WRP10W33 -	97.2%
WRP11W33 -	98.1%

TABLE 6 . INTERPOLYMER FIXED CHARGE MEMBRANE TEST RESULTS

Membrane*	Water flux, m^3/m^2-day (gfd)	Magnesium sulfate rejection,%	Process flux at 3 hrs, m^3/m^2-day (gfd)	Color rejection, %
A	1.54 (37.6)	48	0.63 (15.3)	96.5
B	1.26 (30.8)	50	0.70 (17.0)	97.8
C	1.75 (42.8)	36	0.71 (17.4)	93.3
D	0.54 (13.1)	54	0.42 (10.2)	97.2
E	1.03 (25.1)	35	0.59 (14.4)	90.7
F	0.40 (9.7)**	52	0.52 (12.6)	93.9
G	0.38 (9.3)**	25	†	†

* Designation of membrane as received. See Appendix D.
** Membrane may not have been properly "wet" before testing.
† A mechanical leak in the stirred cell occurred during the test.

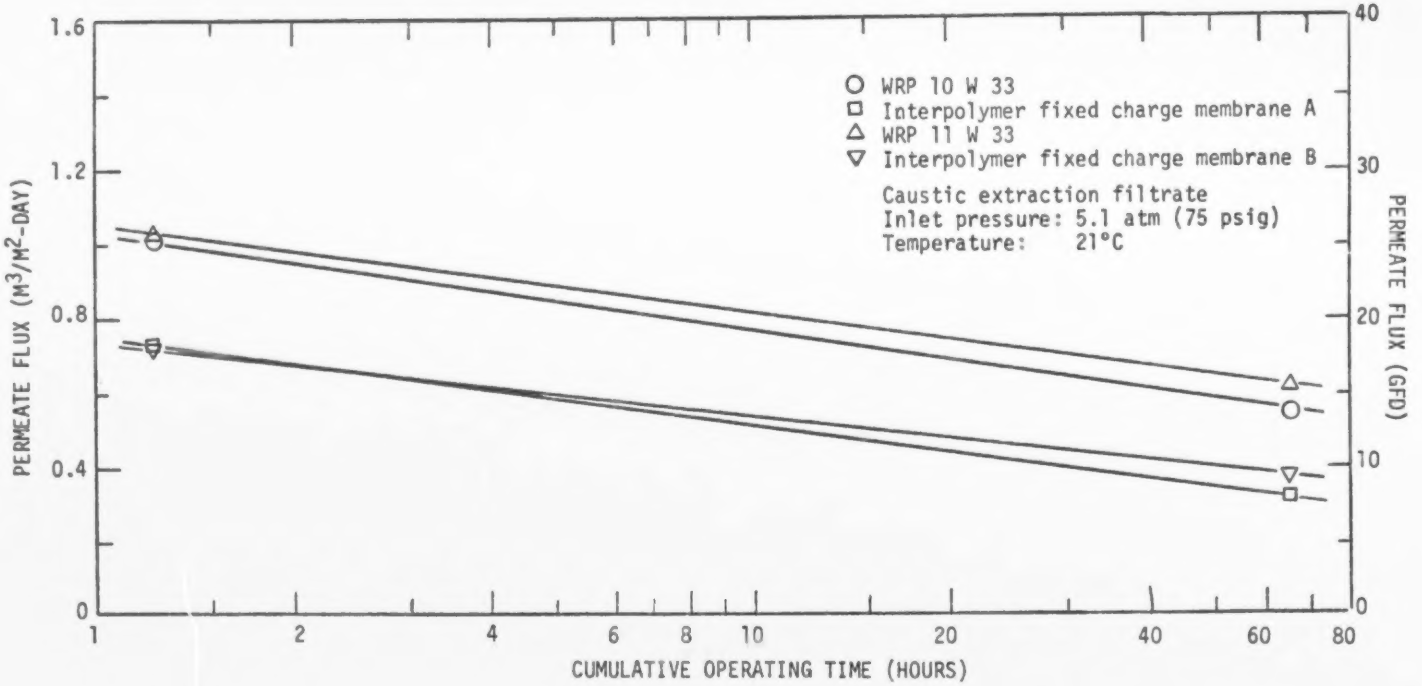


Figure 29. Comparison of interpolymer fixed charge and WRP membrane flux and decline.

Thus the WRP membranes showed higher color rejection and higher flux levels than the interpolymer fixed charge membranes. These superior performance characteristics, coupled with the essentially identical rates of flux decline, indicated that WRP membranes were preferred for color removal applications at Kraft pulp mills.

WRP Spiral-Wound Module Laboratory Test

Based upon the WRP/interpolymer fixed charge membrane test results and the dependence of casting dope viscosity on polymer concentration it was decided to prepare continuous flat sheet membrane from the WRP11W33 formulation. This flat sheet membrane was fabricated into two 0.1 m (4 in) diameter x 0.91 m (36 in) long spiral-wound modules. Each module contained approximately 3.72 m^2 (40 ft^2) of active membrane area.

One module was tested in total recycle with caustic extraction filtrate for 140 hours. The module flux performance during this test is shown in Figure 30. The flux was high, 2.05 to 2.67 m^3/m^2-day (49°C, 4.4 atm, 43.6 m^3/day recirculation flow rate) [50 to 65 gfd (120°F, 65 psig, 8 gpm)], and stable. The higher flux observed in the module as compared to stirred-cell test results is partially a function of increased feed temperature and improved hydrodynamics across the membrane surface.

Two other factors potentially affecting membrane flux performance are changes in membrane properties associated with the adaptation of casting procedures from the laboratory-scale to production-scale, and changes in the feed stream due to aging. These two factors also contributed to a reduction in membrane rejection for color bodies. The one set of samples collected during this run (taken at 24 hours cumulative operating time) showed a module color rejection of only 86% as compared to WRP11W33 flat-sheet color rejection of >97%. The degree to which scale-up and feed degradation each affected membrane performance could not be determined quantitatively. It was assumed, however, that the necessity of shipping the feed solution for this test by truck and the lack of feed refrigeration caused the higher molecular weight color bodies to settle-out resulting in an unrepresentative feed sample*.

At this point laboratory testing was completed. Further evaluation of WRP spiral-wound modules was performed at the Canton Mill where fresh feed would be available on a continuous basis.

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* Samples of caustic extraction filtrate used during stirred-cell tests were air-shipped in 0.02 m^3 (5 gal) drums from Canton, North Carolina and refrigerated upon arrival at the test laboratory.

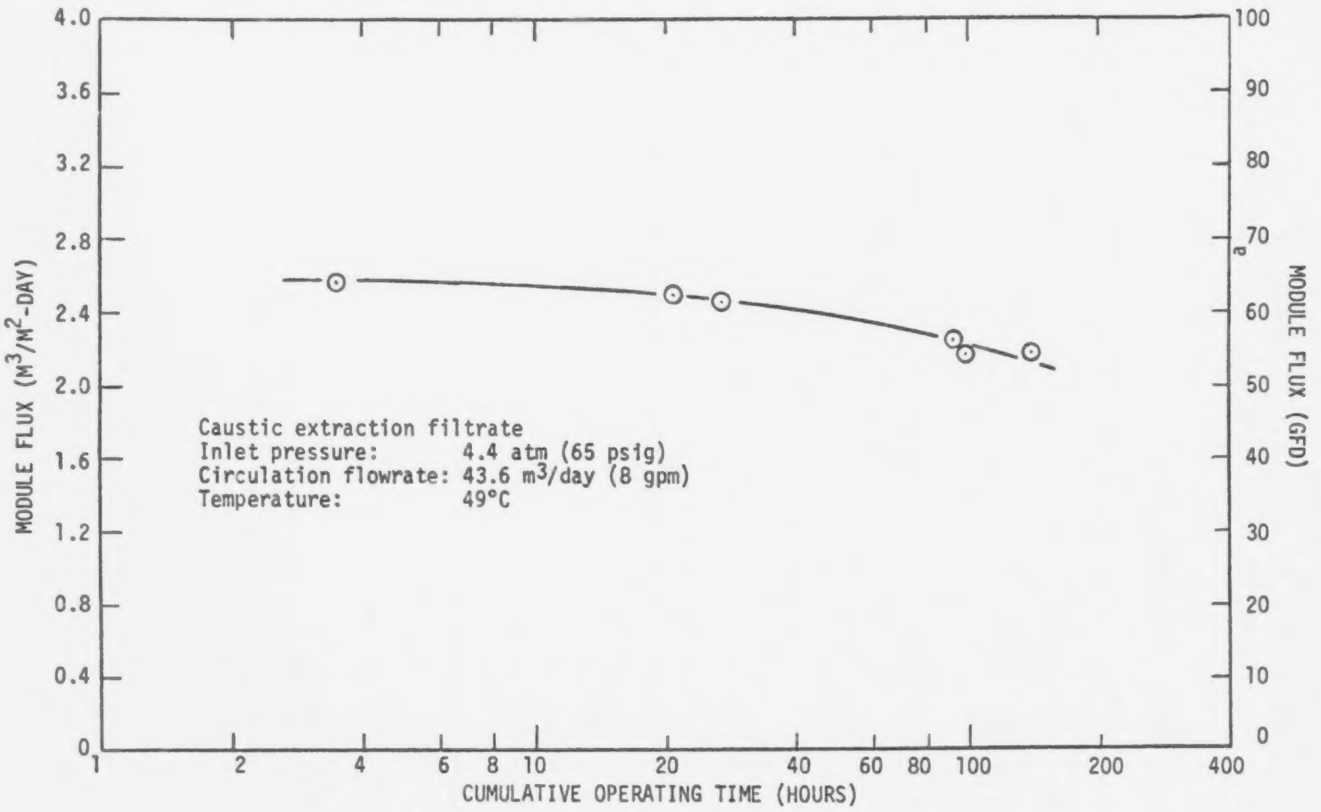

Figure 30. Full-scale Vexar WRP11W33 spiral-wound module flux versus time during laboratory total recycle test.

FIELD EXPERIENCE WITH SPIRAL-WOUND MODULES

Introduction

Before initiating studies with the 3-stage pilot system, testing of single spiral-wound modules was performed. In this manner the effectiveness of various pretreatment options, the preferred conditions for module operation and the degree of module flux recovery following cleaning could be determined in more controlled experiments. Also, a significant level of experience would be gained under actual field conditions while awaiting fabrication of the remaining spiral-wound cartridges. At the conclusion of the single module tests, the 3-stage pilot system was operated for a 3-month period. Essentially all of these tests with spiral modules were performed with the caustic extraction filtrate stream.

Throughout all of the spiral cartridge tests the main indicators of module performance were membrane flux, flux recovery and color rejection.

Single Module Test Results

Six different spiral cartridges were employed during the single module tests. The module designations and their descriptions are listed in Table 7. Both the HR and LR modules became partially dry during shipment and were rewetted by soaking in an isopropyl alcohol and surfactant solution.

Single module experimentation focused on:

- -- membrane flux dependency on time;
- -- effects of feed pH adjustment, feed temperature and inlet pressure on module flux decline;
- -- module pressure drop as a function of time and feed flowrate; and,
- -- module cleaning requirements.

Additionally, sodium hexametaphosphate addition for colloid stabilization was performed, general observations on cartridge mechanical integrity were made and module color rejection was monitored. At this point in the program color rejection was not a major concern since the extensive laboratory screening tests had shown membranes could be "tailored" to this application.

A summary of all single module tests is given in Table 8. Except for a lone 7 hour test (#9) with pine decker effluent all tests were performed with caustic extraction filtrate as the feed stream. All flux data shown in Table 8 have been temperature corrected to 50°C using a ratio of the viscosity of water at the actual process temperature to the viscosity of water at 50°C. The process flux data were generally recorded at 38°C to 50°C; water flux measurements were generally read at 20°C to 40°C. Over a

TABLE 7. MODULES EMPLOYED DURING SINGLE MODULE TESTS

Module designation	Description
HR	WRP11W33, 60 mil vexar spacer; incurred partial drying during shipment.
LR	WRP11W33, 60 mil vexar spacer; incurred partial drying during shipment.
WRP-1	WRP09W33, 60 mil vexar spacer.
WRP-2	WRP09W33, 60 mil vexar spacer.
O-PS	Polysulfone membrane, 30 mil vexar spacer; manufactured by Osmonics, Inc.
O-CA	Cellulose Acetate membrane, 30 mil vexar spacer; manufactured by Osmonics, Inc.

TABLE 8. CHRONOLOGY OF SINGLE MODULE TEST EXPERIENCE

Test no.	Module no.	Test duration, hrs	Initial process flux m^3/m^2-day (gfd)*	Final process flux m^3/m^2-day (gfd)*	Pre-test water flux m^3/m^2-day (gfd)*	Post-test water flux m^3/m^2-day (gfd)*	Range of color rejection, %	Comments
1	HR	20.9	0.78 (19.1)	0.19 (4.6)	2.69 (65.5)	--	50-60	Module changed without final water flux.
2	LR	15.5	0.67 (16.4)	0.21 (5.1)	0.88 (21.4)	0.80 (19.5)	40-50	
3	LR	18	0.95 (23.2)	0.09 (2.1)	0.80 (19.5)	0.55 (13.4)	--	Initial process flux reading at 21°C.
4	HR	6	1.16 (28.4)	0.34 (8.4)	1.78 (43.4)	2.33 (56.9)	56-79	
5	WRP-1	16	4.22 (103)	1.06 (25.8)	4.80 (117)	1.79 (43.6)	72-94	Possible pin-hole leaks in module accounting for high initial flux and low initial rejection.
6	HR	22	1.12 (27.4)	0.14 (3.3)	3.26 (79.6)	1.23 (30.1)	71-94	
7	HR	21.5	0.62 (15.2)	0.09 (2.1)	1.23 (30.1)	0.32 (7.7)	35-85	
8	HR	4.5	0.30 (7.2)	0.23 (5.7)	0.32 (7.7)	0.84 (20.4)	82-90	
9	HR	7	0.45 (10.9)	0.18 (4.3)	0.84 (20.4)	0.95 (23.2)	54-73	Pine decker effluent.
10	HR	22	0.44 (10.8)	0.89 (2.3)	0.95 (23.2)	1.24 (30.3)	65-74	Caustic extraction filtrate adjusted to pH 7.
11	HR	19	0.80 (19.5)	0.34 (8.2)	1.24 (30.3)	0.68 (16.5)	78-92	
12	HR	6.5	0.62 (15.1)	0.34 (8.4)	0.68 (16.5)	2.16 (52.7)	89-92	
13	HR	21.5	0.55 (13.3)	0.23 (5.7)	2.16 (52.7)	1.18 (28.8)	82-87	Caustic extraction filtrate adjusted to pH 7.
14	HR	8	0.76 (18.6)	0.43 (10.5)	1.18 (28.8)	--	88-93	

(continued)

TABLE 8. (CONTINUED)

Test no.	Module no.	Test duration, hrs	Initial process flux, m^3/m^2-day (gfd)*	Final process flux, m^3/m^2-day (gfd)*	Pre-test water flux, m^3/m^2-day (gfd)*	Post-test water flux, m^3/m^2-day (gfd)*	Range of color rejection,%	Comments
15	WRP-2	22	1.30 (31.8)	0.36 (8.7)	1.02 (24.8)	1.79 (43.6)	66-73	Test run at 2.04 atm (30 psig).
16	WRP-2	19.5	1.11 (27.0)	0.40 (9.7)	1.79 (43.6)	--	44-63	Test run at 2.04 atm.
17	WRP-2	21	0.80 (19.6)	0.39 (9.5)	--	--	51-65	Test run at 2.04 atm.
18	WRP-2	4	0.81 (19.7)	0.59 (14.5)	--	--	58-63	Test run at 2.04 atm.
19	LR	5	1.12 (27.2)	0.47 (11.4)	1.53 (37.2)	--	84-90	Test run at 60°C.
20	LR	23	0.96 (23.5)	0.12 (2.9)	--	--	18-75	
21	LR	7.5	2.64 (64.3)	1.19 (29.1)	--	--	(21-24)	Module run in first stage of three stage system Data indicate probable leak.
22	LR	7.5	0.84 (20.4)	0.76 (18.5)	--	--	67-74	Module run in first stage of three stage system.
23	LR	52	0.92 (22.5)	0.32 (7.9)	--	0.59 (14.3)	66-82	Module run in first stage of three stage system.
24	LR	25	0.42 (10.3)	0.19 (4.7)	0.59 (14.3)	--	59-63	Module run in first stage of three stage system. Test run at 60°C. Feed adjusted to pH 12.
25	LR	27	0.58 (14.1)	0.23 (5.6)	--	--	58-76	Module run in first stage of three stage system. Feed adjusted to pH 12.
26	O-PS	23	4.31 (105)	0.60 (14.7)	6.07 (148)	2.38 (58.0)	56-61	
27	O-PS	21	1.99 (48.5)	0.62 (15.0)	2.38 (58.0)	1.32 (32.3)	54	
28	WRP-2	22	2.52 (61.4)	0.61 (14.8)	3.87 (94.5)	2.19 (53.3)	79-86	Module had new outer wrap and new brine seal.
29	O-CA	20	2.35 (57.3)	1.44 (35.0)	2.77 (67.6)	2.43 (59.2)	71-86	Feed adjusted to pH 9 during first 4.5 hours of run. Module rinsed with water 3 times during run.

(continued)

TABLE 8. (CONTINUED)

Test no.	Module no.	Test duration, hrs	Initial process flux, m^3/m^2-day (gfd)*	Final process flux, m^3/m^2-day (gfd)*	Pre-test water flux, m^3/m^2-day (gfd)*	Post-test water flux, m^3/m^2-day (gfd)*	Range of color rejection, %	Comments
30	O-CA	26	2.19 (53.4)	1.24 (30.3)	2.43 (59.2)	1.51 (36.8)	74-79	
31	O-CA	21	1.23 (30.0)	0.40 (9.8)	1.51 (36.8)	1.59 (38.8)	45-84	
32	O-CA	21.5	1.27 (31.0)	0.25 (6.2)	1.59 (38.8)	1.38 (33.6)	53-59	
33	O-CA	17.5	1.05 (25.5)	0.34 (8.3)	1.38 (33.6)	1.64 (39.9)	56-68	
34	WRP-1	19	2.15 (52.5)	0.84 (20.4)	3.94 (96.1)	2.70 (65.9)	86	
35	HR	21.5	2.32 (56.6)	0.29 (7.0)	4.08 (99.6)	--	84	
36	WRP-1	18	2.19 (53.4)	0.62 (15.1)	2.70 (65.9)	3.21 (78.4)	90	
37	WRP-1	3	2.29 (55.8)	1.41 (34.3)	3.21 (78.4)	3.42 (83.5)	79	Test terminated when caustic line went down.
38	WRP-1	22	2.51 (61.3)	0.63 (15.3)	3.42 (83.5)	2.49 (60.7)	85-92	
39	WRP-1	18.5	1.87 (45.6)	0.58 (14.2)	2.49 (60.7)	2.30 (56.0)	79-88	
40	WRP-1	22	1.47 (35.8)	0.52 (12.7)	2.30 (56.0)	3.09 (75.3)	76-87	
41	WRP-1	14.5	2.21 (53.8)	1.15 (28.0)	3.09 (75.3)	3.33 (81.3)	72-81	Test run in total recycle mode.
42	WRP-1	75	1.82 (44.5)	0.55 (13.3)	3.33 (81.3)	2.33 (56.9)	63-83	Test run in total recycle mode. Sodium hexametaphosphate added to caustic extraction filtrate in attempt to stabilize colloidal matter.
43	WRP-1	53	1.31 (32.0)	0.76 (18.5)	2.33 (56.9)	2.62 (61.5)	70-84	Test run in total recycle mode.

* All flux readings temperature corrected to 50°C.

wide temperature range (i.e., 25°C to 50°C) the viscosity-ratio method may be slightly inaccurate; however, for ease of test-to-test data comparison a single reference point (50°C) was deemed desirable.

Using Table 8 for an overview, specific data observations will be discussed below.

Typical Module Flux Performance--

In order to more fully appreciate the effects of the various techniques employed for flux improvement, a "typical" flux decline curve will be examined initially. The flux versus time plot for such a case, test #6, is shown in Figure 31. In this test the HR module processed caustic extraction filtrate on a once-through basis for 22 hours. Initial module water flux was 3.26 m^3/m^2-day (79.6 gfd). Process flux at time zero, 2.37 m^3/m^2-day (57.9 gfd), represents dilution of the caustic stream by water from the backflush depth filter. In 0.3 hours, as the system was purged of water, process flux declined by over 50% to 1.12 m^3/m^2-day (27.4 gfd). The module flux continued to decline rapidly over the next 2 to 3 hours. A leveling off begins to occur at $\sim$0.41 m^3/m^2-day ($\sim$10 gfd). Overnight operation of the system resulted in a gradual loss of flux down to 0.14 m^3/m^2-day (3.3 gfd).

Again, it must be noted that no appreciable concentration occurred during this test: the caustic extraction filtrate was processed on a once-through basis. Also, no increase in pressure drop was observed across the module. The flux decline is therefore related to operating time rather than concentration effects (increased solids loading) or module plugging by fibers (reduced membrane area and lower average pressure).

A flux pattern of the nature observed in test #6 indicates a steady fouling of the membrane surface by some specie(s) in the caustic extraction filtrate stream. At this point in the program the foulant(s) had not been identified. It was clear, however, from both technical and economic standpoints that either process operating conditions had to be modified or additional feed pretreatment exercised if satisfactory flux levels were to be achieved and maintained.

Total Recycle Flux Performance--

The flux vs. time curves for two total recycle tests (tests #41 and 43) with the WRP-1 module are given in Figure 32. In both instances approximately a 40% flux decline occurred during the first 5 hours of processing. The flux loss tapered-off slightly between 5 and 10 hours processing and then stabilized at 1.15 m^3/m^2-day (28 gfd) and 0.82 m^3/m^2-day (20 gfd) for tests #41 and 43, respectively. By contrast, the "typical" once-through process test (see Figure 31) showed nearly an 80% flux loss in the first 5 hours with a subsequent steady flux decline down to 0.12 m^3/m^2-day (3 gfd).

Operation of a single module in total recycle at the laboratory had shown stable flux performance on the order of 2.05 m^3/m^2-day (50 gfd) (see Figure 30), whereas total recycle operation in the field resulted in stable

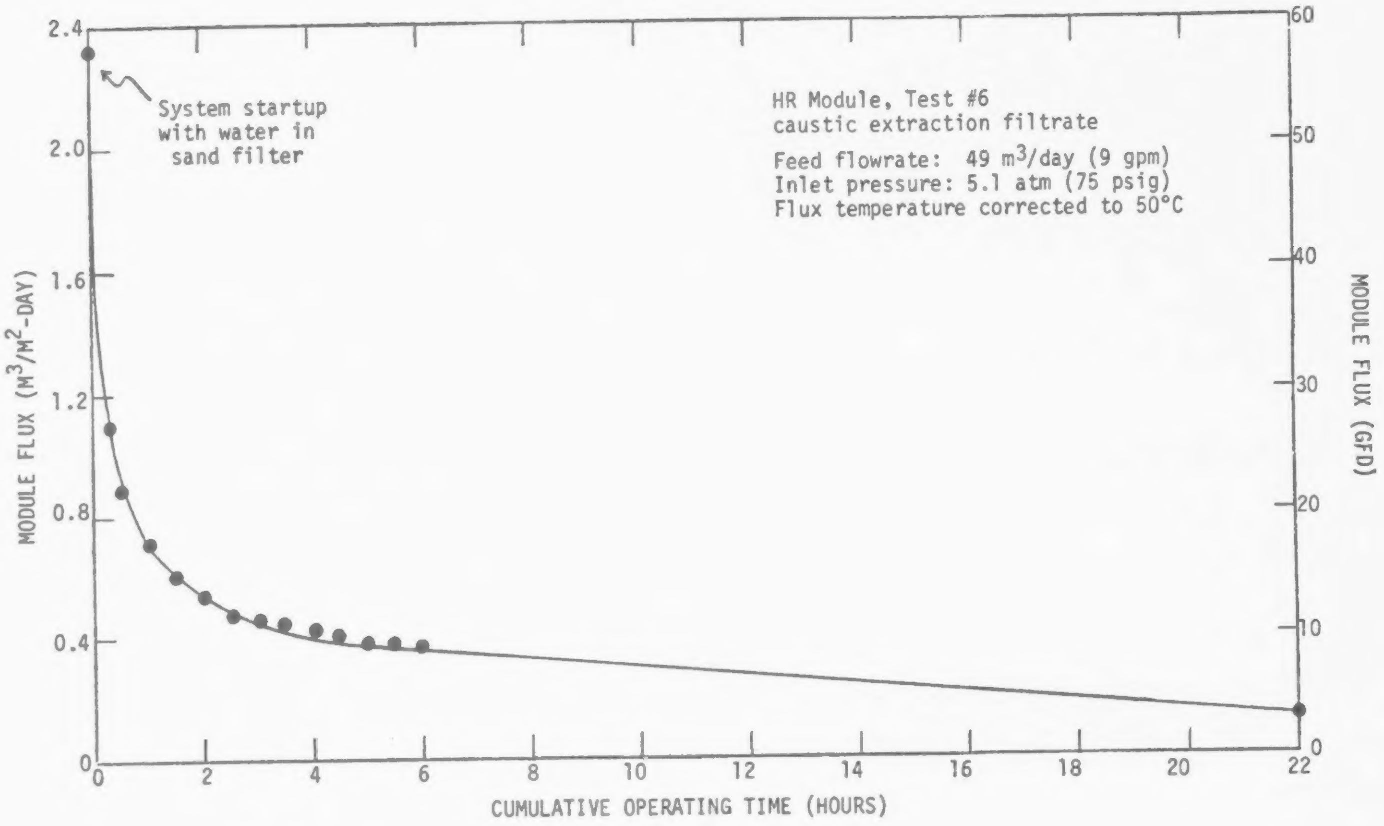

Figure 31. "Typical" module flux performance during single module tests.

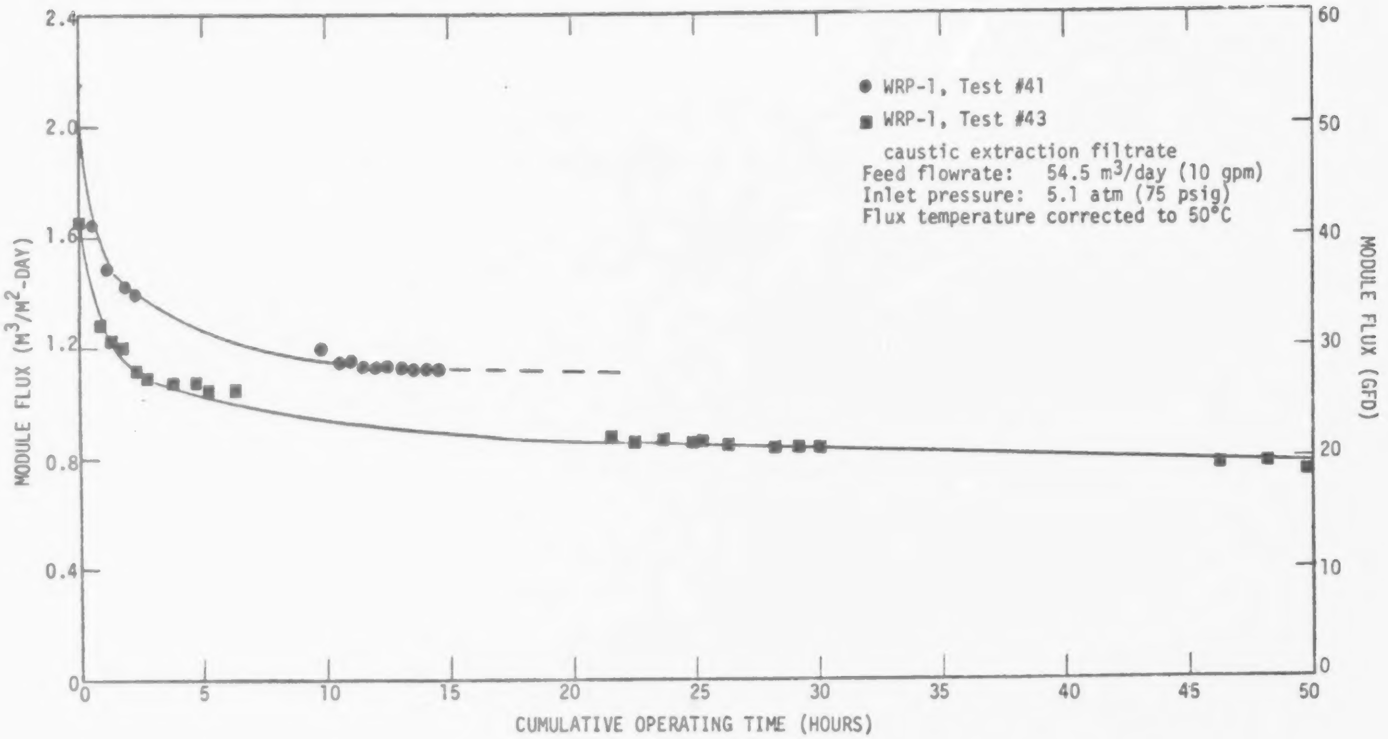

Figure 32. Module flux performance during total recycle tests.

module flux only after initial steep flux declines. Changes in the feed stream (i.e., aging, changing mill conditions) are believed to account for these different flux patterns.

The stabilization of module flux in the total recycle mode suggested a finite quantity of membrane foulant(s) was present in the caustic extraction filtrate. Once the fairly rapid foulant/membrane interaction was complete, steady flux was maintained. Again, control of the foulant(s) on a continuous basis was required.

Module Flux with pH Adjusted Feed--

The average pH of the caustic extraction filtrate was 10.8. Upward adjustment of the pH could potentially stabilize colloids in the feed, while downward pH adjustment was expected to agglomerate colloidal matter. In the latter case, some fraction of the suspended solids would be removed by the system prefilters.

Figure 33 shows the flux versus time curves for tests at pH 7 (tests #10 and 13) and at pH 12 (test #25). In all three experiments initial process flux was somewhat low (0.41 to 0.82 m^3/m^2-day) [10 to 20 gfd]. While this may be partially due to low pre-test water flux levels in tests #10 and 25, the pre-test water flux of 2.16 m^3/m^2-day (52.7 gfd) in test #13 belies complete dependence on this factor.

For the tests at pH 7 a 50% flux loss occurred within 5 hours operating time. This is similar to the results of the total recycle tests. From this point the flux decline was slow, but constant, with final flux readings of 0.08 to 0.25 m^3/m^2-day (2 to 6 gfd).

The test conducted at pH 12 indicated slightly less severe fouling with a 32% flux decline in 5 hours. After five hours, however, the slope of the flux versus time curves for the pH 12 and pH 7 tests were essentially identical.

In summary, no noticeable improvement in module flux performance resulted from feed pH adjustment.

Module Flux at Elevated Temperature--

The transport of water (flux) across a membrane surface increases with increasing temperature. Increased process temperature has also been shown to alter the fouling characteristics of certain feed streams (35). Thus, the caustic extraction filtrate stream was processed at 60°C in two single module runs (tests #19 and 24). In test #24 the feed pH was also adjusted upward to pH 12.

The duration of test #19 was only 5 hours, however in that time a 58% flux decline occurred. Test #24, conducted for 25 hours, had an initial module flux of only 0.5 m^3/m^2-day (12.1 gfd) (60°C) and a final flux level

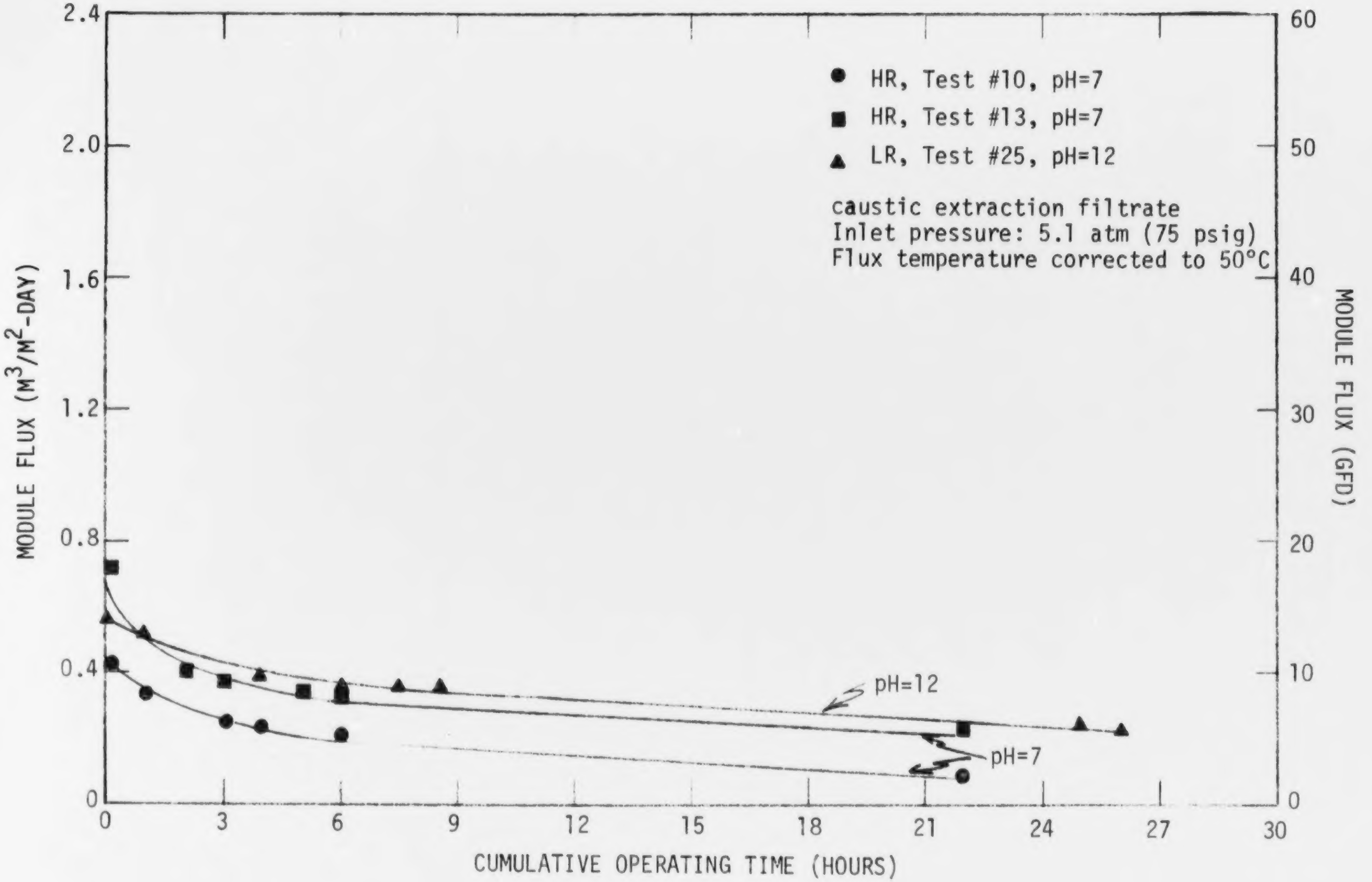

Figure 33. Module flux performance during pH adjustment tests.

of 0.23 m^3/m^2-day (5.5 gfd). Based on the results of these two experiments processing at elevated temperature does not reduce membrane fouling and, hence, is not recommended.

Module Flux with Sodium Hexametaphosphate Addition--

One experiment was performed with sodium hexametaphosphate (SHMP) added to the feed to reduce the precipitation of colloids onto the membrane surface. The SHMP was added to the caustic stream at a 10 ppm concentration and a 75 hour total recycle test was conducted (test #42). Within 5 hours a 54% flux loss, from 1.82 to 0.84 m^3/m^2-day (44.5 gfd to 20.5 gfd), was observed. Thus the addition of SHMP had no effect on the startup performance of the module. After 30 hours total recycle the flux stabilized at 0.53 to 0.62 m^3/m^2-day (13 to 15 gfd) for the duration of the test. Since the SHMP showed no initial flux improvement, stability probably resulted from the total recycle nature of the test rather than from colloid stabilization.

Module Flux with Lower Inlet Pressure--

Membrane compaction is a common, and sometimes highly significant, phenomenon in the operation of reverse osmosis membrane modules. While ultrafiltration is performed at much lower pressures than reverse osmosis, the potential exists for UF membrane compaction leading to flux decline and inability to recover initial module flux. To investigate this possibility four tests (Tests #15 through 18) were conducted with the WRP-2 module operated at 2 atm (30 psig) inlet pressure. This module had not been operated at all prior to these tests.

The flux history of WRP-2 during the low pressure tests is shown in Figure 34. In each of the four tests significant flux loss is observed in the initial portion of the test. The sharp flux decline is followed by a gradual drop in flux throughout the remainder of each test. These results are similar to the flux patterns developed at 5.1 atm (75 psig) inlet pressure. It can also be noted from Figure 34 that detergent cleanings could not recover process flux to its initial value (1.3 m^3/m^2-day) [32 gfd]. In fact, a 40% decline in initial process flux is observed by the fourth test.

The membrane fouling occuring during caustic extraction filtrate processing thus appears independent of inlet pressure in the range of 2 to 5.1 atm (30 to 75 psig). As such, membrane compaction is not suspected to be of any significance for this application.

Comparison of Individual Module Flux Performances--

In addition to varying operating conditions and feed pretreatments to isolate the factors contributing to poor membrane flux performance, modules other than the WRP series were investigated. In this manner it could be determined if the major foulant(s) were preferentially affecting the WRP-type membrane. The membrane modules evaluated as alternatives to the WRP modules were polysulfone and cellulose acetate modules commercially available from Osmonics, Inc. These modules were designated O-PS and O-CA,

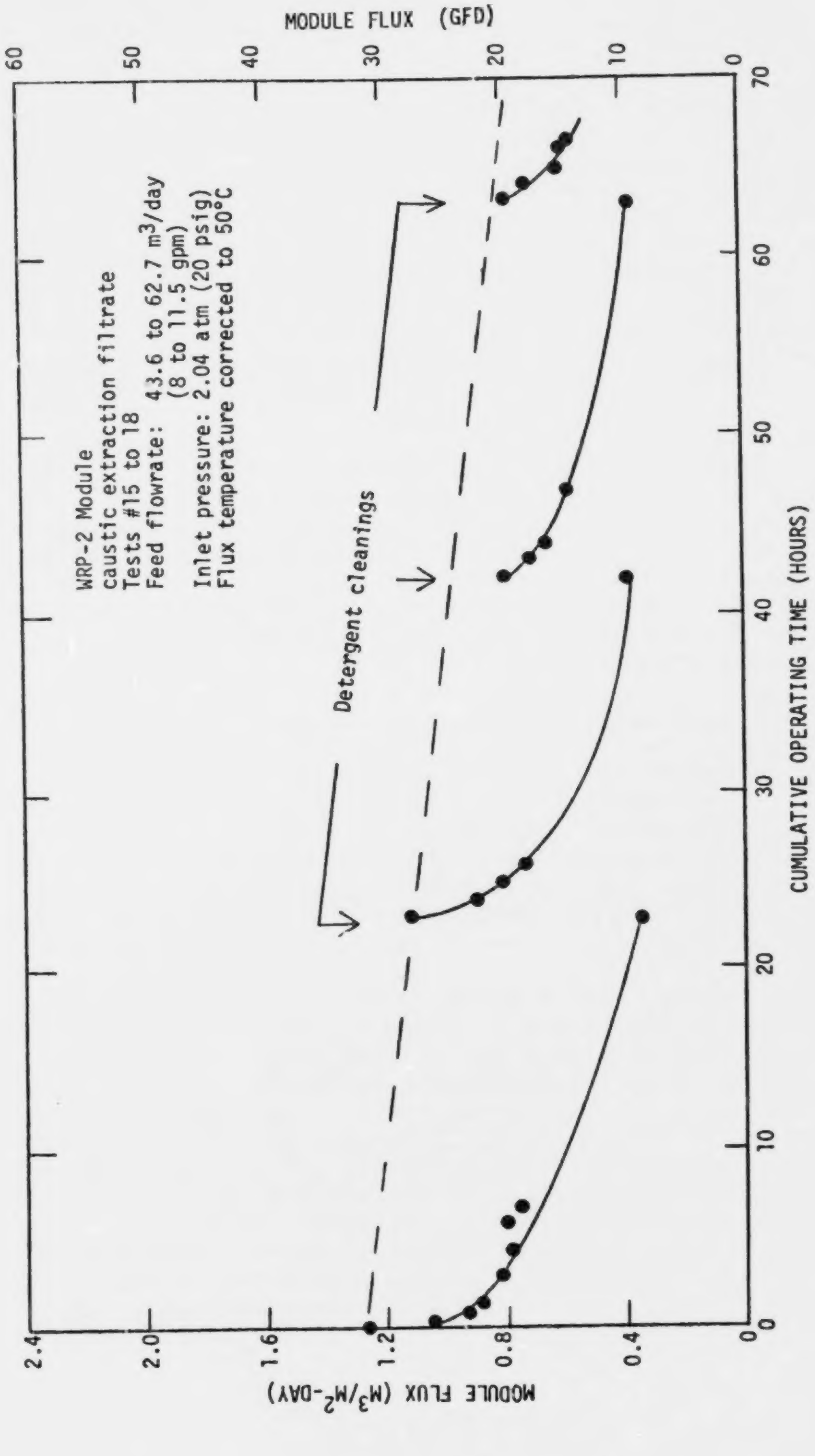

Figure 34. Module flux performance at low inlet pressure (2.04 atm) [30 psig].

respectively. The CA module was operated initially with a pH 9 feed stream to hydrolyze the cellulose acetate to cellulose. It was then expected that the O-CA module could be operated at the natural pH and temperature of the caustic extraction filtrate.

Flux decline is plotted in Figure 35 as a function of operating time for the WRP-1, O-PS and O-CA modules. Even though the individual tests were performed over a several month period almost identical flux decline curves were obtained. Thus the membrane fouling and module flux decline are not specific to the WRP spiral-wound modules but to other polysulfone-based modules and cellulosic modules, as well.

Module Pressure Drop Considerations--

The measurement of pressure drop across a spiral-wound cartridge is important for two reasons. First, an increase in pressure drop with time (at constant flowrate) signals module plugging. This could indicate incomplete pretreatment, especially in streams containing fibrous material. Second, the power requirement for an ultrafiltration system is determined almost entirely by the power input to the feed circulation pump. This power input is directly proportional to the product of the volumetric output of the pump and the pressure drop across the module system. An increase in circulation rate causes a concomitant increase in pressure drop, greatly affecting system power requirements. Therefore, a tradeoff exists between improved flux and increased power costs as the circulation rate is raised.

Plugging of a spiral-wound module can have several deleterious effects: reduction in effective membrane area due to feed channel blockage; lower average driving pressure across the module, possibly reducing flux; and reduction in module flux recovery with cleaning, leading to shortened module life. In this program, no statistically significant data were obtained on module pressure drop versus time because of mechanical problems (brine seal failures), module changeover during the tests and use of uncalibrated pressure gauges. However, the daily log sheet data suggested no build-up of pressure drop with operating time. This is most likely the case since inspection of the feed channel spacer within one module showed no entrapped fiber or other solids. On this basis the pretreatment sequence, while not able to reduce module fouling, was sufficient to prevent module plugging.

Module flux versus pressure drop (plotted as flux versus circulation rate which was more accurately measured) is shown in Figure 36 for HR module processing of caustic extraction filtrate over a 7 day period. The extreme scatter in the data and the lack of a linear relationship (log-log plot) indicate flux sensitivity to circulation flow is minimal. Thus, in the normal operating range of Vexar-spacer spirals (27.3 to 81.8 m^3/m^2-day recirculation [5 to 15 gpm] no improvement in flux with increased feed superficial velocity is observed.

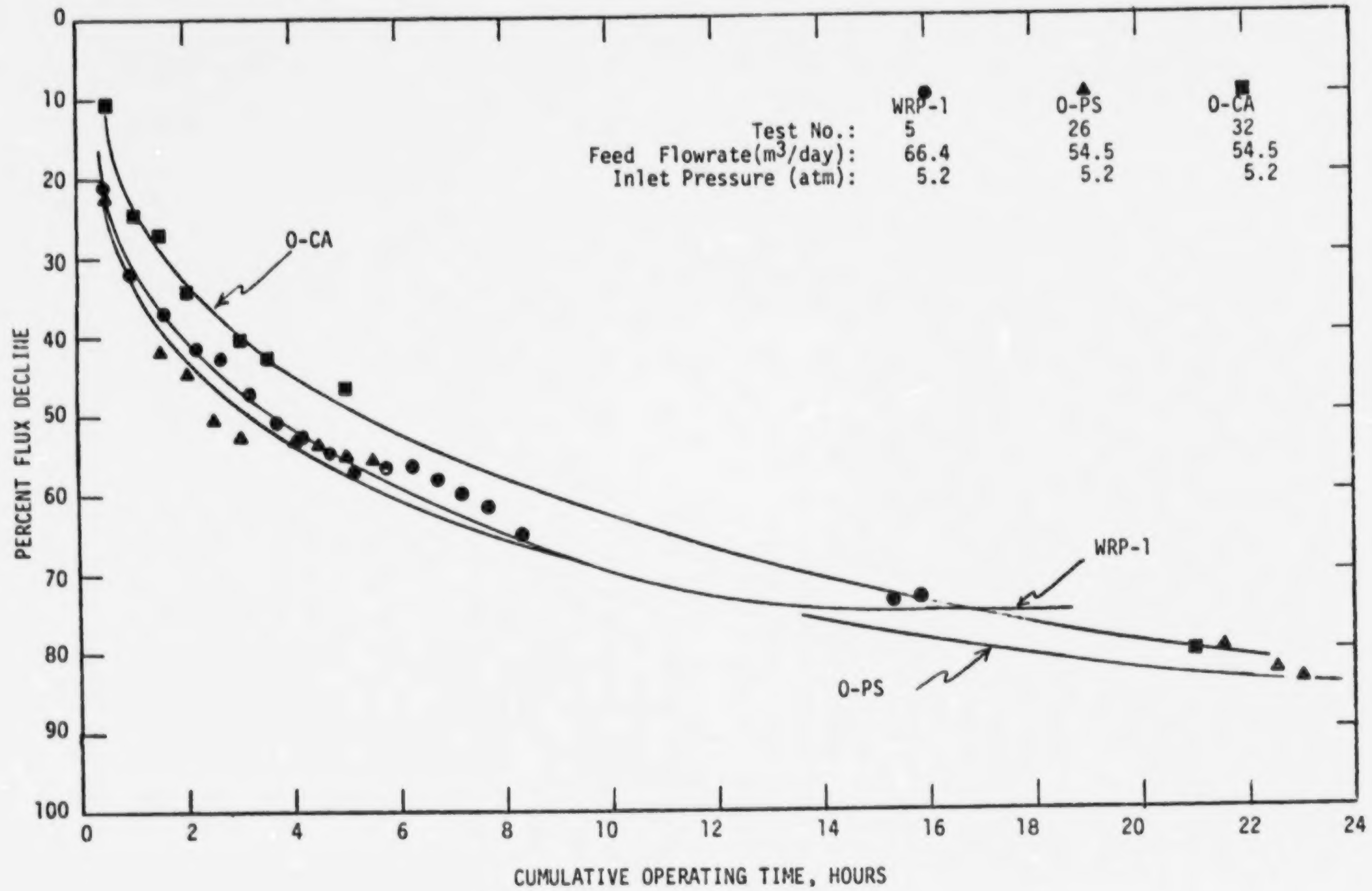

Figure 35. Module flux decline as a function of operating time.

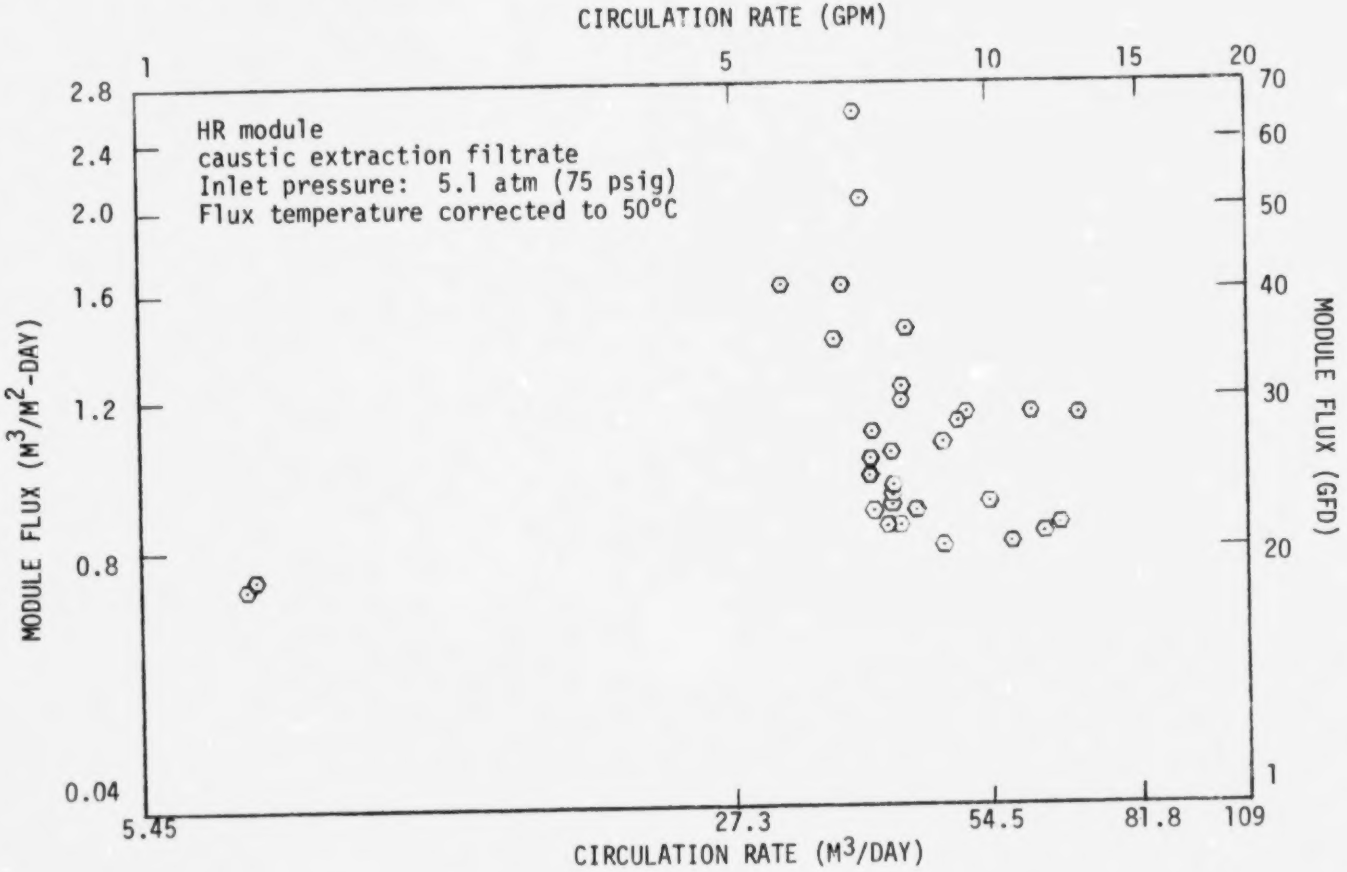

Figure 36. Module flux versus circulation rate.

In summary, neither increased pressure drop at constant flow (plugging) nor improved flux at increasing flow (reduced concentration polarization) were observed during the single module tests.

Module Mechanical Problems--

The mechanical integrity of the spiral-wound modules was generally good but two problems did occur with prolonged operation. One problem was that initially the resin impregnated fiberglass outer wrap on the modules became loose and could easily slide off. If not protected by a strong outer wrap the module could be punctured or damaged during handling. This problem was overcome by changing to an epoxy which was more chemically stable than the original impregnating resin. Unfortunately, this improved resin must be heat cured presenting the possibility of membrane drying during fabrication.

The second mechanical problem was brine seal failure. The seals lost strength, occasionally tore and generally degenerated to the point where feed flow bypassed the face of the module. Under these conditions pressure drop measurements became meaningless and actual circulation rates through the module questionable. No successful solution to this problem was identified.

Module Flux Recovery--

As observed in Table 8 the spiral-wound modules could generally be cleaned. What cannot be discerned from this Table is the difficulty encountered in the cleaning operations and the unpredictability of the results. The occasionally week-long cleaning cycles that were needed are clearly impractical for large-scale system operation. It is because of the difficulty of module flux recovery that the single module tests were extended for the major portion of this program. In this way both cleaning procedures and pretreatment techniques could be further investigated.

The cleaning procedures employed were described previously. The discussion of cleaning effectiveness will be deferred until all membrane flux data have been presented.

Module Color Rejection--

Color rejection exhibited by the spiral modules was lower than would have been expected based on the laboratory screening test data. In fact, it was seldom that module color rejection exceeded 90% (see Table 8). Because of the significant flux decline problems which occurred and the emphasis placed on feed pretreatment and foulant(s) identification, improved module color rejection was not sought. The experimental design was to concentrate on module flux improvement first. Then, if warranted, continuous flat sheet WRP membrane could be recast with better color rejection characteristics and fabricated into spiral-wound modules.

Pretreatment Testing and Foulant Identification Summary

Analysis of a fouled membrane surface produced the following analytical results:

Volatiles at 105°C	88.0%
Kaolinite clay	5.6%
Starch	4.0%
Titanium dioxide	1.4%
Carboxylic acid salt	<1.0%

The major components of the fouling "slime" layer thus come from recycle of white water from the paper mill back to the pulp mill. It is now known when mill operators began to recycle white water. However, now that this water conservation measure is in effect, the pulp mill ultrafiltration system must be capable of treating a stream containing both clay and TiO_2.

Foulant removal was attempted with

- -- Various depth filter media;
- -- Chemical coagulant addition followed by depth filtration; and,
- -- Chemical coagulant addition followed by vacuum filtration.

The chemical coagulants tested (mainly in jar tests) were Nalco coagulants GWP-827, 7132, 107, and 8103; Chitosan; Arquad 2HT75; animal glue; lime and acid addition with the Nalco polymers.

No satisfactory pretreatment method was identified during this program. Detailed results of the pretreatment studies can be found in Appendix E.

It is of note that when one module was cut open for examination the slime layer was readily removed from the membrane surface by simple wiping. This indicates that the foulants were not bonded to the membrane but were rejected in the gel layer adjacent to the membrane surface.

Three-Stage Pilot System Test Results

An effective pretreatment technique had not been identified by start-up of the 3-stage pilot system. Nonetheless, the staged-pilot system was operated for 335 hours over a 3-month period. It was hypothesized that the first stage modules might be preferrentially fouled by the white water species, thereby limiting fouling in the second and third stages. If this phenomenon occurred, meaningful flux data at concentrated feed levels could be obtained.

As was the case with the single module tests, emphasis was placed on module flux and flux recovery rather than color rejection. The feed stream to the staged pilot plant was caustic extraction filtrate for all but a 9-hour period. During this brief time when the caustic line was down, pine decker effluent was processed.

Narrative of Pilot Plant Flux Performance--

Discussion of pilot plant flux performance will proceed chronologically through the 335 hours of system operation. To aid discussion, flux versus time data for the 3 system stages are plotted in Figures 37, 38, 39, and 40. Typical overall system conversions are indicated in the Figures. Routinely 95% to 97% conversion (20 to 30-fold volume reduction) was maintained.

Pilot system startup was with one module per stage. During the first 90 hours (see Figure 37) only limited flux data were available for stages 1 and 2 as permeate production was below scale on the rotometers. Initial process flux levels were 0.82 to 1.0 m^3/m^2-day (20 to 25 gfd) for all three stages. The stage 1 and 2 flux levels dropped off scale rapidly while the stage 3 flux followed the pattern observed in the single module tests: rapid initial flux loss then a more gradual decline over the next 20 hours. A single 2.5 hour caustic/EDTA wash was performed after 25 hours exposure to the waste stream. Based on third stage process flux of 0.62 m^3/m^2-day (15 gfd), the cleaning was only partly successful.

A much more gradual decline in process flux is observed in the next processing period: 26 to 48 hours. Here a 12-hour wash encompassing 2 caustic/EDTA cycles and a 4.5 hour ultraclean cycle, recovered stage 2 and 3 water flux (darkened symbols) to acceptable levels. Reintroduction of the caustic extraction filtrate brought stage 3 flux quickly to 0.51 m^3/m^2-day (12.5 gfd). Flux then gradually declined to approximately 0.41 m^3/m^2-day (10 gfd). An ultraclean/caustic wash at this point was ineffectual, limiting process data over the subsequent 10 hour period.

After a total of 81 operating hours the stage 3 module was removed and stages 1 and 2 received a 2-hour ultraclean/EDTA wash. Flux recovery was 0.62 m^3/m^2-day (15 gfd) for stage 1 and 0.49 m^3/m^2-day (12 gfd) for stage 2.

Six hours following return to the process stream, stage 2 flux began to rise and its permeate became very dark in color. This module was removed and replaced. No module failure autopsy was performed.

Pilot system operation continued (see Figure 38) with the original module in stage 1, a new module (previously in stage 3) now in stage 2 and no modules in stage 3. Two ultraclean washes (one with EDTA) produced flux levels of 0.90 m^3/m^2-day (21.9 gfd) and 1.76 m^3/m^2-day (43.0 gfd) in stages 1 and 2, respectively. An uninterrupted 44-hour processing period showed continually decreasing flux for both stages with the stage 1 permeate output approximately 50% below that of stage 2. This inversion may indicate greater deposition of white water foulants on the stage 1 membrane surface.

A 2-hour ultraclean/EDTA wash brought stage 1 flux to 0.41 m^3/m^2-day (10.0 gfd) and stage 2 flux to 1.21 m^3/m^2-day (29.5 gfd). Process flux immediately fell to <50% of the water flux for both stages and another 2-hour ultraclean wash was initiated. Flux recovery for stage 1 improved slightly to 0.49 m^3/m^2-day (12 gfd); for stage 2 only 0.82 m^3/m^2-day

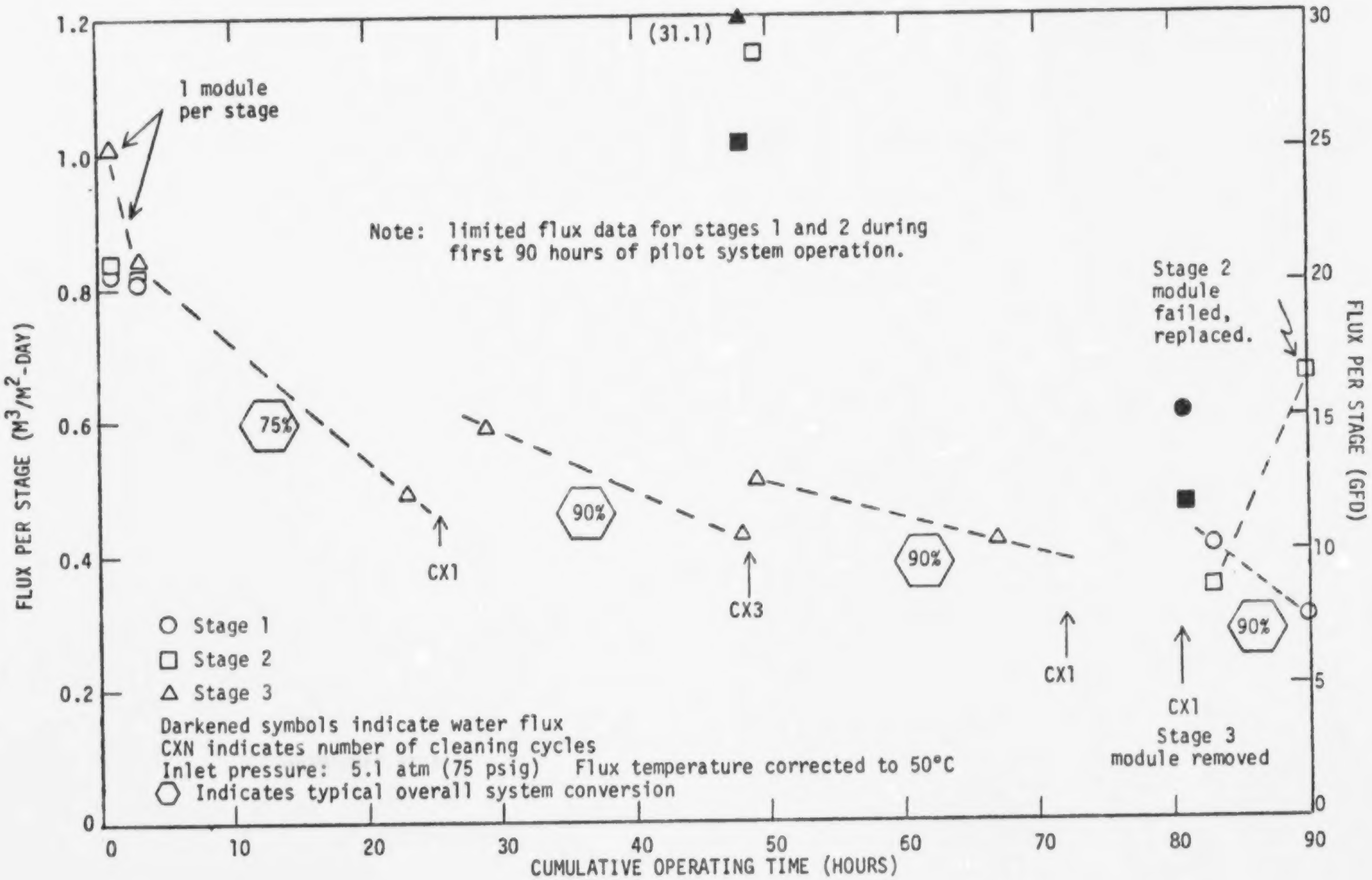

Figure 37. Flux history during 3-stage pilot system operation (0 to 90 hours).

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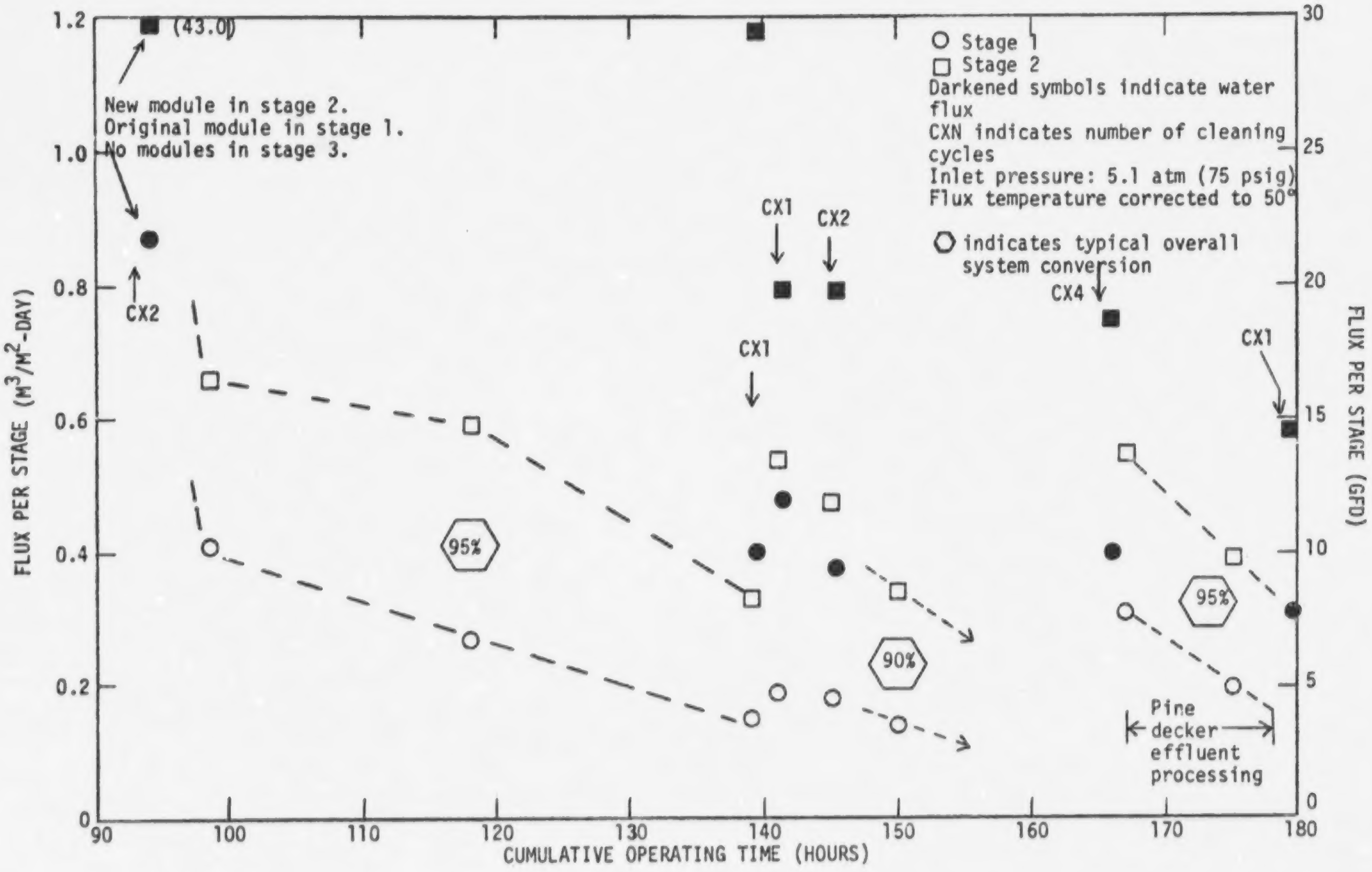


Figure 38. Flux history during 3-stage pilot system operation (90 to 180 hours).

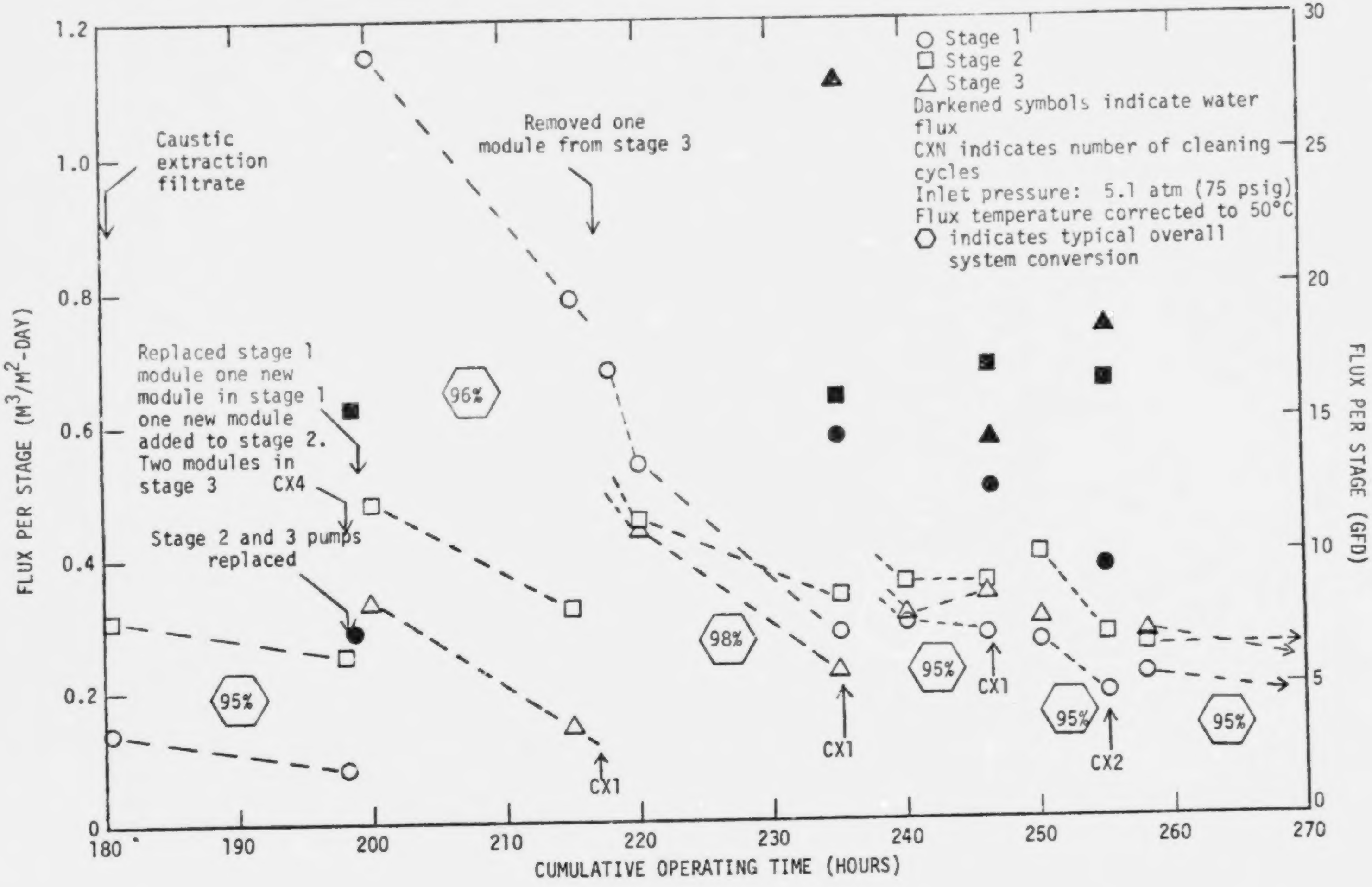


Figure 39. Flux history during 3-stage pilot system operation (180 to 270 hours).

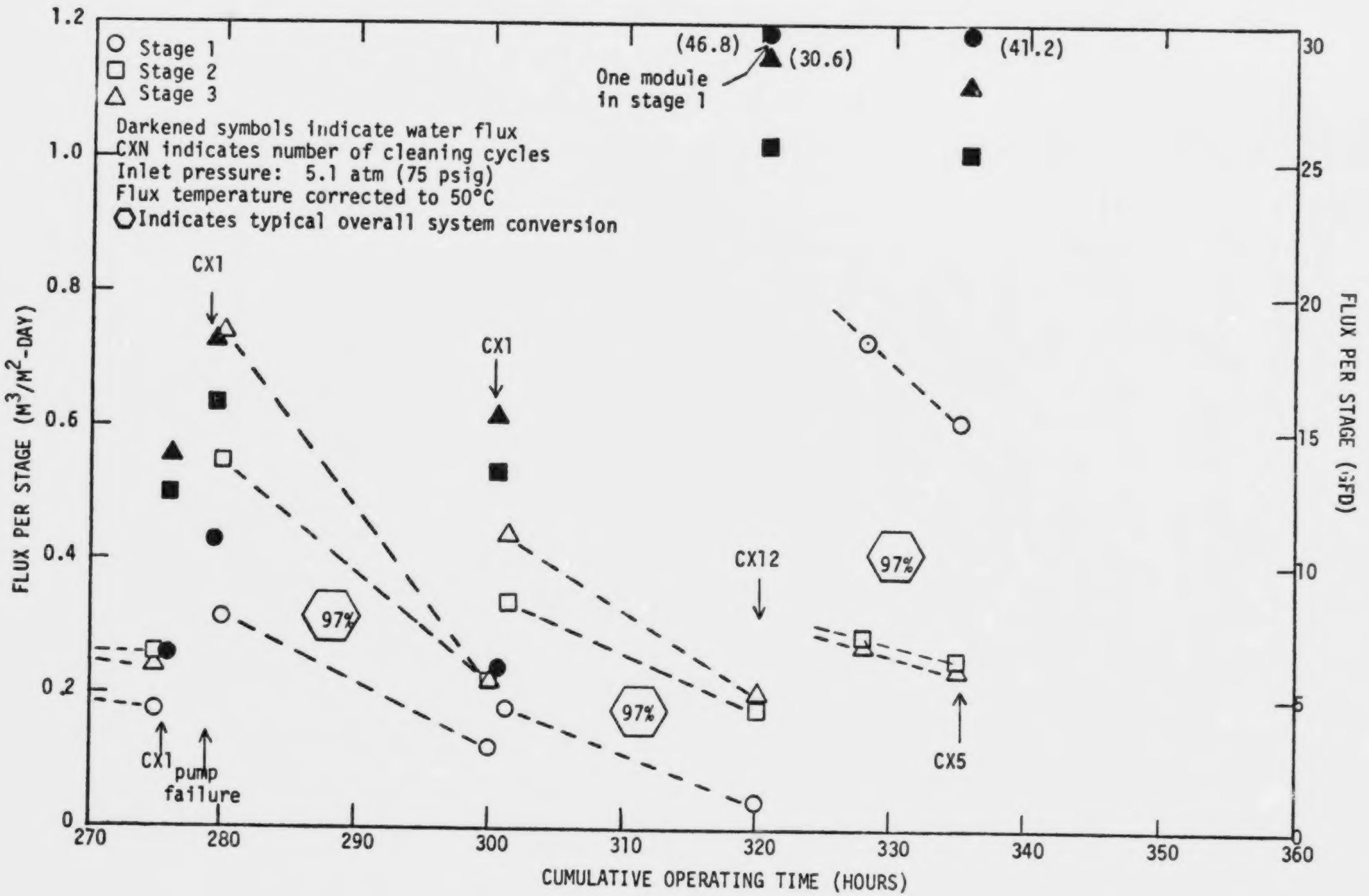


Figure 40. Flux history during 3-stage pilot system operation (270 to 335 hours).

(20 gfd) was recorded. Process flux for stage 1 was below 0.21 m^3/m^2-day (5 gfd) within a few hours after washing necessitating a further cleaning step. This time two ultraclean/EDTA cycles (4.5 hours total duration) were employed. Essentially no change in either stage water flux was observed.

Process flux for both stages began at <0.41 m^3/m^2-day (10 gfd) and continued to decline over the next 20-hour period. Two ultraclean cycles (2.5 hours) and two ultraclean/EDTA cycles (6 hours) recovered stage 1 water flux to only 0.41 m^3/m^2-day (10 gfd) and stage 2 water flux to 0.77 m^3/m^2-day (18.7 gfd).

Pine decker effluent was now processed as the caustic extraction filtrate line was down. The flux pattern remained unchanged and a single 2.5 hour ultraclean/EDTA wash produced the typically low water flux levels.

A return to caustic extraction filtrate showed routine flux losses over the next operating period (180 to 197 hours, see Figure 39). By the conclusion of this processing period final flux for stage 1 was only 0.09 m^3/m^2-day (2.1 gfd). System operation was now interrupted as new circulation pumps were installed in the second and third stages. Three ultraclean washes and one ultraclean/EDTA wash (9.5 hours, in total) were then performed with flux recovery to 0.30 m^3/m^2-day (7.3 gfd) for stage 1 and 0.64 m^3/m^2-day (15.6 gfd) for stage 2.

The stage 1 module, which had experienced nearly 200 hours of exposure to the mill effluents appeared to be irreverisbly fouled and was replaced with a new module. The pilot system was now operated with 1 module in stage 1, 2 modules in stage 2 and 2 modules in stage 3. The next 20 hours operation period began with process flux levels of 1.18 m^3/m^2-day, 0.50 m^3/m^2-day, 0.34 m^3/m^2-day (28.8 gfd, 12.1 gfd, and 8.3 gfd) for stages 1, 2 and 3, respectively. Flux for all stages declined but the final stage 1 flux was still relatively high (0.80 m^3/m^2-day) [19.6 gfd].

After a 1.5 hour ultraclean wash, one module was removed from stage 3 and the system returned to caustic extraction filtrate processing. For all stages process flux began below 0.74 m^3/m^2-day (18 gfd) and measured 0.21 to 0.41 m^3/m^2-day (5 to 10 gfd) within 15 hours.

The subsequent 3 processing periods showed less than 0.41 m^3/m^2-day (10 gfd) flux for each stage. Also flux recovery was consistently worse for stage 1 than for stages 2 and 3.

A 2-hour ultraclean wash cycle was conducted both before and after a pump failure at the 279-hour marks (Figure 40). Resultant process flux at each stage exhibited some initial improvement but after 20 hours was 0.12 to 0.25 m^3/m^2-day (3 to 6 gfd) for all stages. A single 2-hour ultraclean wash followed, producing low water fluxes. Subsequent process flux declined typically.

A series of 12 wash cycles, as listed in Table 9, recovered overall system water flux to 1.42 m^3/m^2-day (34.6 gfd). These wash cycles were mainly performed with ultraclean and totaled 85.5 hours exposure of membrane to cleaning solutions.

It was noticed at this time that some improvement in water flux occurred when one, rather than two modules was present in a housing. A check of pressure drop at various flow rates through both a single module and a pair of modules in series produced the expected results. This ruled out the possibility of seal failure in the second modules allowing feed to bypass the membrane surface and giving an overall lower flux. While this problem was discussed extensively, it was not resolved.

A final 20-hour processing period was initiated with stages 2 and 3 process flux once again in the 0.21 to 0.41 m^3/m^2-day (5 to 10 gfd) range. The ending water flux values, after 4 ultraclean washes (28 hours) and 1 caustic/EDTA wash (4.5 hours) were 1.69 m^3/m^2-day (41.2 gfd) for stage 1, 1.04 m^3/m^2-day (25.3 gfd) for stage 2 and 1.14 m^3/m^2-day (27.9 gfd) for stage 3.

Summary of Pilot Plant Flux Performance--

The several hundred hour pilot plant test reconfirmed the position that spiral-wound module systems - modules and pretreatment - are not currently suitable for integrated kraft mill effluent processing. In non-integrated mills, where the pulping operation is independent of the paper making process, severe membrane fouling may not occur since white water will be absent. During this program, however, there was no mechanism for segregating the bleachery and decker effluents from white water recycle to test this hypothesis.

General conclusions which can be drawn from 3-stage pilot plant operation on caustic extraction filtrate are:

-- At constant conversion, module flux declines rapidly (within 24 hours) to economically unacceptable flux levels.

-- Except in instances where new modules were installed in a particular stage, flux levels per stage were not significantly different after a few hours operating time. No stage was able to maintain a flux rate of >0.61 m^3/m^2-day (15 gfd) and typically performance was 0.21 to 0.41 m^3/m^2-day (5 to 10 gfd).

-- There is some evidence to suggest that the first stage modules are more heavily fouled than the subsequent stages. Flux recovery for the first stage being consistently below that for stages 2 and 3.

-- System flux and flux decline were basically unchanged during a brief processing period with pine decker effluent.

-- Individual module exposure time to the waste stream had a greater influence on module flux than the degree of individual stage or overall system conversion.

TABLE 9 . SEQUENCE OF CLEANING SOLUTIONS APPLIED TO 3-STAGE PILOT SYSTEM AFTER 320 HOURS OPERATING TIME

Cleaning solution	Cleaning duration, hrs	Overall system water flux, m^3/m^2-day (gfd)
1. Caustic/EDTA*	2.25	-- --
2. Ultraclean/enzyme	1.75	0.32 (7.9)
3. Ultraclean	15.75	0.65 (15.8)
4. Ultraclean	1.5	-- --
5. Caustic/EDTA	3.0	0.80 (19.6)
6. Ultraclean	16.5	0.78 (19.0)
7. Ultraclean/enzyme	2.0	-- --
8. Ultraclean/EDTA	3.0	0.82 (19.9)
9. Ultraclean	14.75	0.89 (21.6)
10. Ultraclean/enzyme*	4.0	0.91 (22.3)
11. Ultraclean	3.5	0.96 (23.5)
12. Ultraclean	17.5	1.04 (25.4)
Double brine seals placed on each module, only one module in stage 1		1.42 (34.6)

* Stage 1 backwashed with ultraclean after this cleaning cycle.

- -- No consistently effective cleaning solution was identified.
- -- Daily cleaning appears necessary. The duration of each cleaning cycle would be a minimum of 2 hours, most likely significantly longer.

It is also important to note that no increase in pressure drop across a stage was observed. This indicates module plugging was not a factor in determining system performance.

Pilot Plant Color Rejection--

Color concentrations of the feed into the pilot system (after the Broughton filters), the recycle stream within each step, the reject stream from each stage and the permeate stream from each stage were routinely recorded during the 335 hours of testing. However, the low rejection performance of the spiral-wound modules (typically 70% to 90%) during the single module tests foretold that meaningful color rejection data would not be obtained during 3-stage pilot plant operation.

The complete color rejection data set is presented in Appendix F and summarized in Table 10. The average feed color concentration was 18,000 c.u. Based on the feed, average rejections were 67.5% for stage 1, 63.6% for stage 2 and 34.6% for stage 3. These values are quite low and would not be acceptable for full-scale operation. Even on a concentrate basis (rejection based on the stream the modules are actually exposed to) rejections were only 73.6%, 84.7% and 91.1% for stages 1, 2 and 3, respectively.

FIELD EXPERIENCE WITH TUBULAR ASSEMBLIES

Introduction

The inability to remove stream foulants to levels suitable for successful spiral-wound module operation led to an investigation of polysulfone membranes in a tubular geometry. The basic considerations which identified tubular assemblies as a viable alternative to spiral-wound modules were:

- -- Higher superficial velocity over the membrane surface is achievable with the tubular configuration. This leads to more turbulent flow and could minimize the gel concentration layer at the membrane/liquid interface.
- -- Tubular assemblies are more readily cleanable than spiral-wound modules, again because of increased feed superficial velocity. Also, tubular assemblies can be cleaned by mechanical means (sponge ball circulation through the tubes), if necessary.

Three forms of tubular assemblies were evaluated:

- -- 12.7 mm (0.5 in) diameter tubes;
- -- 12.7 mm (0.5 in) diameter tubes with volume displacers; and
- -- 25.4 mm (1 in) diameter tubes.

TABLE 10. SUMMARY OF 3-STAGE PILOT SYSTEM COLOR REJECTION AND PERMEATE QUALITY DURING CAUSTIC EXTRACTION FILTRATE PROCESSING

Stage	Average color concentration (color units)				Average rejection (%)	
	Feed stream	Recycle stream	Reject stream	Permeate stream	Feed basis	Concentrate (recycle stream) basis
1	18,000	24,200	28,200	6,080	67.5	73.6
2	18,000	50,100	61,400	7,210	63.6	84.7
3	18,000	159,000	206,000	16,300	34.6	91.1

The 12.7 mm (0.5 in) diameter tubes were tested first since they could be readily bob-cast and then inserted in epoxy-reinforced fiberglass support tubes. Successful operation of these tubes made change over of the 3-stage pilot system to tubular modules of prime interest. To accomplish such a change over rapidly, and within prevailing budgetary constraints, required use of existing pumps and piping. This, in turn required tubular modules with turbulence promoting spheres since they could be operated at lower flowrates than open-channel tubes. Before fabrication of tubular modules with turbulence promoters, this concept was tested at the Canton Mill to determine if its flux characteristics paralleled those of the open-channel 12.7 mm (0.5 in) diameter tubular membranes. Finally, the development of polysulfone casting solutions which could be cast directly onto fiberglass supports allowed the evaluation of 25.4 mm (1 in) diameter tubular assemblies.

12.7 mm Diameter Tubular Assemblies

General--

Four 12.7 mm (0.5 in) x 1.2 m (4 ft) long tubular membranes were prepared from the same WRP membrane formulation developed for flat sheet casting. Tubular assemblies were made by inserting the membranes in support tubes and securing them with grommets and expanders. The developmental nature of tubular polysulfone casting led to wide variation in the individual tube flux and rejection characteristics. Initial performance data for these tubes is given in Table 11. Water flux ranged from 1.0 to 5.49 m^3/m^2-day (24.6 to 134 gfd) (50°C) and total solids rejection ranged from 43% to 86%.

The single module test stand was modified to run the tubular assemblies and the 4 tubes were connected in series; tube M4 in the lead position. Because of either improper membrane insertion or jarring during shipment, the M4 membrane was stripped from its backing with the initial flow of water. This tube was removed, tube C2 was stored as a spare, and the test stand was restarted with tubes C5 and 01 connected in series. These tubes were operated for nearly 500 hours with no further mechanical problems occurring.

Performance Characteristics--

Figure 41 shows individual tube flux and color rejection as functions of operating time throughout their field evaluation. Also plotted is concentrate flow rate versus time. Since the caustic extraction filtrate was processed on a once-through basis, concentrate flow is equivalent to feed circulation rate (i.e., a measure of feed superficial velocity).

Flux for the two tubes was vastly different: 0.62 to 0.82 m^3/m^2-day (15 to 20 gfd) for tube 01, 1.64 to 2.26 m^3/m^2-day (40 to 55 gfd) for tube C5. These data follow directly from the wide difference (3.28 m^3/m^2-day) [80 gfd] in water flux for the 2 tubes. The flux trends for the 2 tubes are, however, identical. An initially sharp decline over the first 20 hours waste exposure followed by a stabilization of flux for the remainder of the test (472 hours).

During the entire processing period, which stretched over 50 days, the system was shut down many times. Following each shut down the system was flushed with water. At no time was detergent or mechanical cleaning employed.

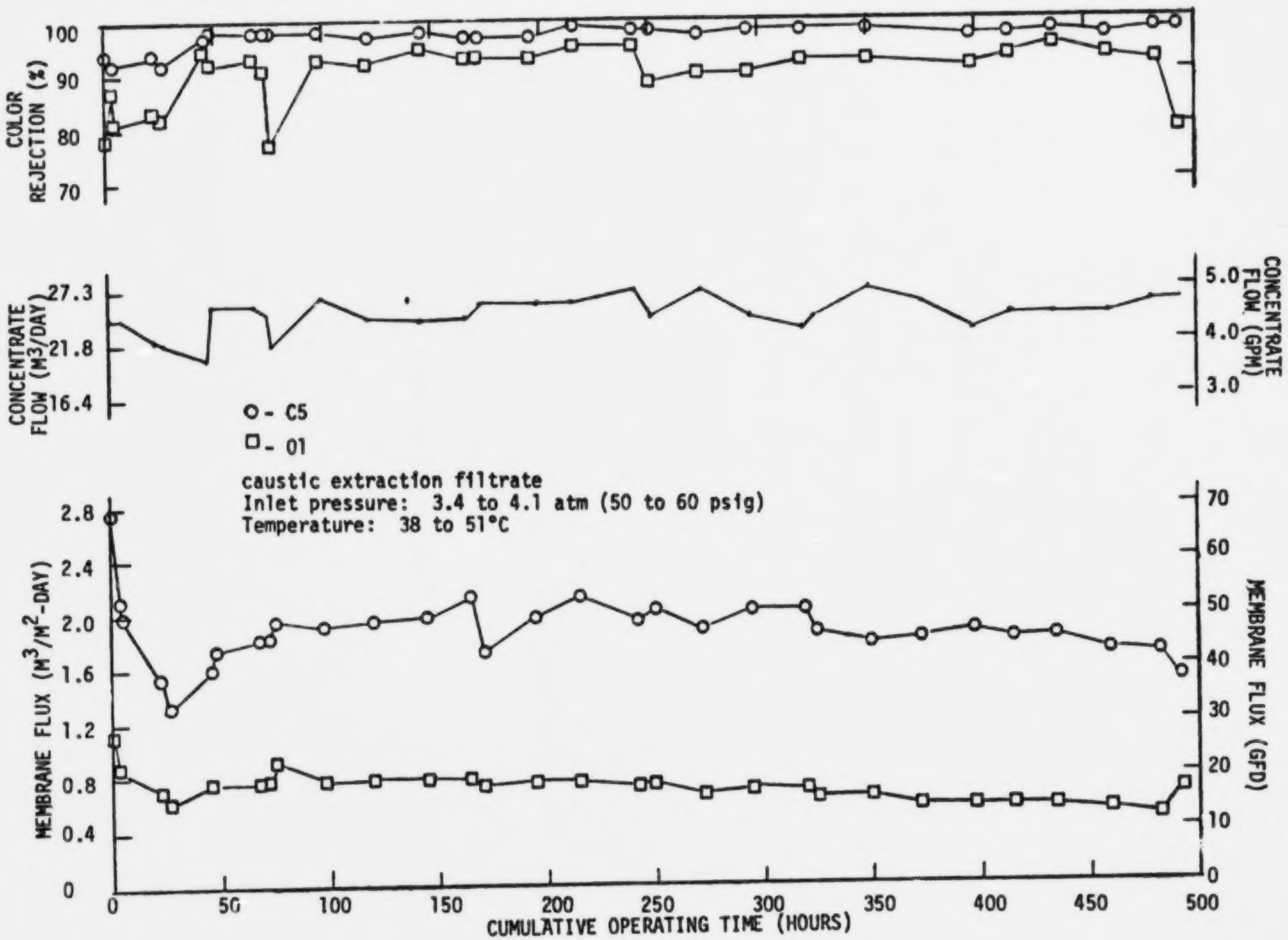


Figure 41. Performance characteristics of 12.7 mm diameter WRP membrane assemblies.

TABLE 11. INITIAL PERFORMANCE CHARACTERISTICS OF 12.7 MM DIAMETER WRP TUBULAR ASSEMBLIES*

Membrane designation	Water flux @ 50°C, m^3/m^2-day (gfd)	Total solids removal efficiency**,%
C2	5.49 (134)	43.2
C5	4.88 (119)	53.6
M4	1.01 (24.6)	85.5
O1	1.47 (35.9)	65.2

* Operating conditions were: Feed circulation rate: 16.4 to 21.8 m^3/day (3 to 4 gpm)

Inlet pressure: 6.1 atm (75 psig)

** Feed solution for removal efficiency measurement was a 2 wt. % solution of Carbowax 6000M.

The average flow through the membranes was 24.5 m^3/day (4.5 gpm). This corresponds to a superficial velocity of 2.26 m/sec (7.4 ft/sec). It can be hypothesized that the flux decline at the start of the run (0 to 25 hours) is associated with the drop in circulation rate below 21.8 m^3/day (1.98 m/sec) [4 gpm (6.5 ft/sec)] and a build up in the membrane gel layer. This theory does not hold, however, since the flux recovered at the 25 hour mark while the flowrate continued to decline (through 46 hours). Hence, if a critical feed superficial velocity (below which fouling occurs) exists with tubular assemblies it was not identified during this test.

Color rejections for the two tubular assemblies were not uniform. The high flux tube, C5, showed 91% to 99% color rejection averaging 97% to 98% rejection. The lower flux 01 tube color rejection varied from 77% to 95%. Thus the WRP tubular assembly color rejection was superior to that of the spiral wound modules in both cases. Also, the exceptional color rejection of the C5 assembly equalled laboratory screening test results with this formulation polysulfone.

12.7 mm Diameter Tubular Assemblies with Turbulence Promoters

It was of interest to test the 12.7 mm (0.5 in) WRP tubes with turbulence promoters because the pumping capacity of the staged pilot system was too low for open-channel tubular modules. The type of module available would have had too high a pressure drop at flows of 21.8 m^3/day (4 gpm) and greater. Tubes packed with turbulence promoting spheres require lower circulation rates to achieve turbulent flow. If successful operation was realized, the pilot system could be outfitted with packed tubular modules without major modification.

Two 12.7 mm (0.5 in) x 0.61 mm (2 ft) tubes with turbulence promoters were prepared. While it is difficult to calculate superficial velocity levels in a packed tube, experience has shown that a flow rate of 2.73 to 5.45 m^3/day (0.5 to 1 gpm) is comparable to a 24.5 to 27.3 m^3/day (4.5 to 5 gpm) open-channel flow rate.

Table 12 shows typical data for the tests with packed tubes. The total flow rate to the system started at 4.9 m^3/day (0.9 gpm) and during the 30 hour test declined to 2.73 m^3/day (0.5 gpm). The pressure drop across the 2-tube module became so high that the flow could not be kept at its initial level. Initially the pressure drop was 3.8 to 3.9 atm (56 to 58 psig). Ending pressure drop was 4.6 to 4.8 atm (68 to 70 psig). Since at the lower flowrate a lower pressure drop is expected, plugging of the packed tube was evident and the test was terminated.

Even with this plugging problem, flux decline was not as severe as with spiral-wound modules.

Color rejection for both WRP tubes was 88% to 93%.

TABLE 12. PERFORMANCE CHARACTERISTICS OF WRP TUBULAR ASSEMBLIES WITH TURBULENCE PROMOTERS

Cumulative operating time, hours	Inlet pressure, atm (psig)	Pressure drop, atm (psig)	Circulation flow rate, m^3/day (gpm)	Flux @ 50°C, m^3/m^2-day (gfd)		Color rejection (%)	
				Tube 1	Tube 2	Tube 1	Tube 2
0	5.2 (76)	3.9 (58)	4.9 (0.90)	--	--	--	--
0.6	5.0 (74)	3.8 (56)	4.8 (0.88)	1.60 (39.0)	0.69 (17.0)	89	88
1.6	5.0 (74)	3.8 (56)	4.7 (0.87)	1.64 (39.9)	0.64 (15.6)	--	--
2.6	5.1 (75)	3.9 (58)	4.7 (0.86)	1.60 (39.0)	0.62 (15.0)	90	89
3.6	5.2 (76)	4.5 (66)	4.6 (0.85)	1.60 (39.0)	0.56 (13.6)	--	--
4.6	5.2 (76)	4.5 (66)	4.4 (0.80)	1.60 (39.0)	0.46 (11.3)	92	89
5.6	5.2 (76)	4.5 (66)	4.2 (0.77)	1.54 (37.5)	0.56 (13.6)	--	--
6.6	5.2 (76)	4.5 (66)	4.1 (0.75)	1.57 (38.4)	0.60 (14.7)	--	--
7.6	5.2 (77)	4.5 (66)	4.1 (0.75)	1.54 (37.5)	0.77 (18.7)	--	--
23.3	5.4 (80)	5.0 (73)	3.3 (0.60)	1.23 (30.0)	0.52 (12.7)	93	92
24.3	5.4 (79)	4.7 (69)	3.3 (0.60)	1.23 (30.0)	0.52 (12.7)	--	--
25.3	5.4 (79)	4.7 (69)	3.3 (0.60)	1.20 (29.3)	0.49 (12.0)	--	--
26.3	5.2 (77)	4.6 (67)	3.2 (0.58)	1.20 (29.3)	0.49 (12.0)	93	91
27.3	5.2 (77)	4.6 (67)	3.1 (0.57)	1.17 (28.5)	0.49 (12.0)	--	--
28.3	5.3 (78)	4.6 (68)	3.1 (0.56)	1.17 (28.5)	0.49 (12.0)	93	92
29.5	5.3 (78)	4.6 (68)	2.9 (0.53)	1.11 (27.0)	0.43 (10.6)	--	--
30.3	5.4 (80)	4.8 (70)	2.9 (0.53)	1.11 (27.0)	0.46 (11.3)	--	--

25.4 mm Diameter Tubular Assemblies

General--

The development of 25.4 mm (1 in) diameter x 1.52 m (5 ft) long tubular polysulfone membranes by Abcor occurred during the final months of this program. These membranes were cast from a proprietary formulation not of the WRP-series. Two membrane assemblies were tested, in series, on caustic extraction filtrate at 3 concentration levels. The concentration levels, simulating individual stage conversions, were:

Concentration Factor	Conversion
1.2X	16.7%
10X	90%
50X	98%

Each concentration level was maintained for about a 1-week period. During the 10X and 50X runs low-molecular weight cut-off HFM tubular assemblies were operated in series with the polysulfone tubes. These membranes were "tighter" than standard HFM membranes but unlike the membranes used in the screening tests did not have any coating treatments.

Performance Characteristics--

Flux--Tubular polysulfone membrane flux versus time is plotted in Figure 42 for the 1.2X (16.7% conversion) test period. The two tubular assemblies showed similar flux patterns over the 132 hour run even though one tube (tube B) consistently had 0.41 to 0.62 m^3/m^2-day (10 to 15 gfd) higher flux than the other tube (tube A).

After a 10% flux loss in the first 5 hours of the run, flux stabilized for 15 hours at 27.5 m^3/m^2-day (67 gfd) for tube A and 3.65 m^3/m^2-day (89 gfd) for tube B. Flux declined gradually over the next 55 hours to 2.71 m^3/m^2-day (54 gfd) for tube A and 2.79 m^3/m^2-day (68 gfd) for tube B. This flux decline is associated with increased total solids levels (see Figure 42). In the final 60 hours of the run very stable flux was achieved except for one unexplained flux increase between the 75 and 85 hour mark. Once stabilized average flux levels were 2.26 m^3/m^2-day (55 gfd) and 2.87 m^3/m^2-day (70 gfd) for tubes A and B, respectively.

During the 1.2X concentration test the feed circulation rate was varied between 109 and 136 m^3/day (20 and 25 gpm). This resulted in a change in feed superficial velocity from 3.1 m/sec (136 m^3/day) [10.2 ft/sec (25 gpm)] to 2.49 m/sec (109 m^3/day) [8.17 ft/sec (20 gpm)]. The first time the feed velocity was lowered permeate flux declined slightly (see Figure 42). Upon increasing feed velocity again flux stabilized. A return to the lower feed velocity for the final 20 hours of the test produced no effect on flux stability. Overall minimal flux loss is observed at the lower flowrate, however further tests will be necessary to fully document the effect of lower circulation rates.

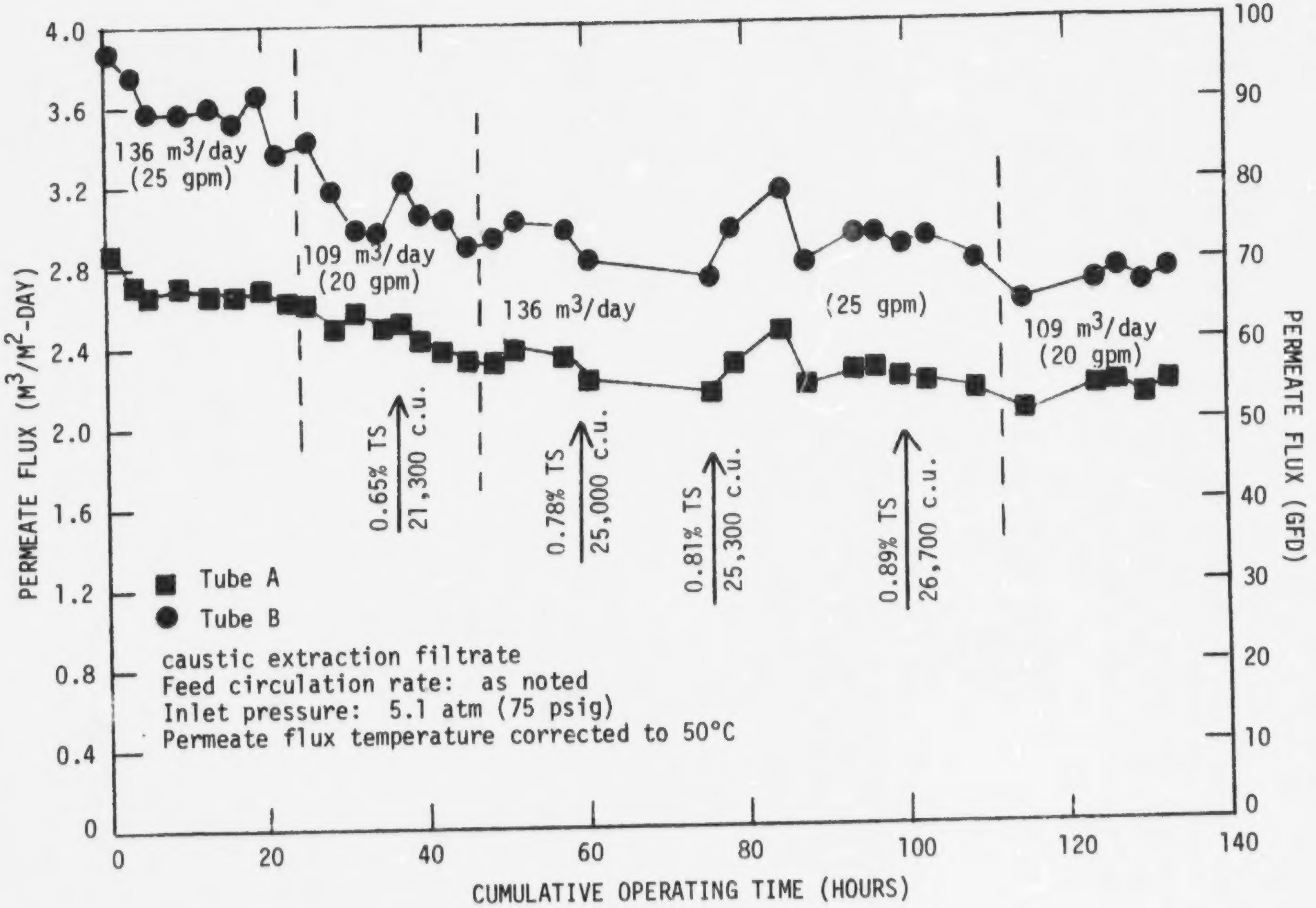


Figure 42. 25.4 mm diameter tubular polysulfone membrane flux versus time during 17% conversion test period.

At both 109 and 136 m^3/day (20 and 25 gpm) the feed superficial velocity is greater than the 2.26 m/sec (7.4 ft/sec) employed in the 12.7 mm (0.5 in) diameter tubular tests. Thus, it may be possible to lower the circulation rate still further without significant flux loss or membrane fouling resulting. This is a very important consideration since system power requirements are greatly increased at higher flowrates. In fact, a reduction of 27.3 m^3/day (5 gpm) in feed circulation from 136 m^3/day (25 gpm) to 109 m^3/day (20 gpm) would result in greater than a 20% decrease in ultrafiltration system pumping power.

Polysulfone membrane flux at a 10X concentration factor (90% conversion) is shown as a function of time in Figure 43. Again the two tubes showed slightly different flux levels while following similar flux curves. Gradual flux loss is observed as the concentration within the membrane loop is allowed to reach 10X. After this point, as a steady conversion was maintained, the flux stabilized at 2.05 m^3/m^2-day (50 gfd) for tube A and 2.49 m^3/m^2-day (60 gfd) for tube B. These flux levels were stable for the final 45 hours of the test.

The 50X concentration test flux data are shown in Figure 44. Tubes A and B exhibited essentially identical flux during this run. Therefore, for clarity, the average flux of the two tubes is plotted in Figure 44. Flux declined gradually over the 180 hour concentration period from 2.87 to 1.03 m^3/m^2-day (70 to 25 gfd). As the concentration was held steady at 50X for the next 90 hours the permeate flux stabilized between 0.94 to 1.07 m^3/m^2-day (23 to 26 gfd).

Low molecular weight cut-off HFM tubular assemblies were run in series with the polysulfone tubes in the tests at 90% and 98% conversion. Figure 45 shows the flux data for the 90% conversion run. One tube, tube D, failed within 24-hours and was removed. Tube C was operated for the full 135 hour test. Its initial flux was 9.02 m^3/m^2-day (220 gfd). This declined sharply to 4.1 m^3/m^2-day (100 gfd) within 20 hours, stabilized for nearly a day's time then began a constant, gradual decline. Final flux was 1.7 m^3/m^2-day (42 gfd), approximately 30% below the average polysulfone membrane flux during the same test.

HFM flux data during the 98% conversion test are plotted in Figure 46. Flux began at 2.05 m^3/m^2-day (50 gfd) and declined to essentially zero in 200 hours. At this point no further HFM flux data were collected.

Flux Recovery--Table 13 summarizes polysulfone tubular membrane flux recovery. After both the 1.2X and the 10X runs (133 and 268 cumulative operating hours, respectively) water flux was recovered to at least initial values with only a 2-hour ultraclean wash. The membranes were not washed at the Canton Mill following the 50X concentration run. Rather, they were partially filled with water, sealed and returned to Abcor. These membranes were not used again for 3 months. At that time they received a 1-hour

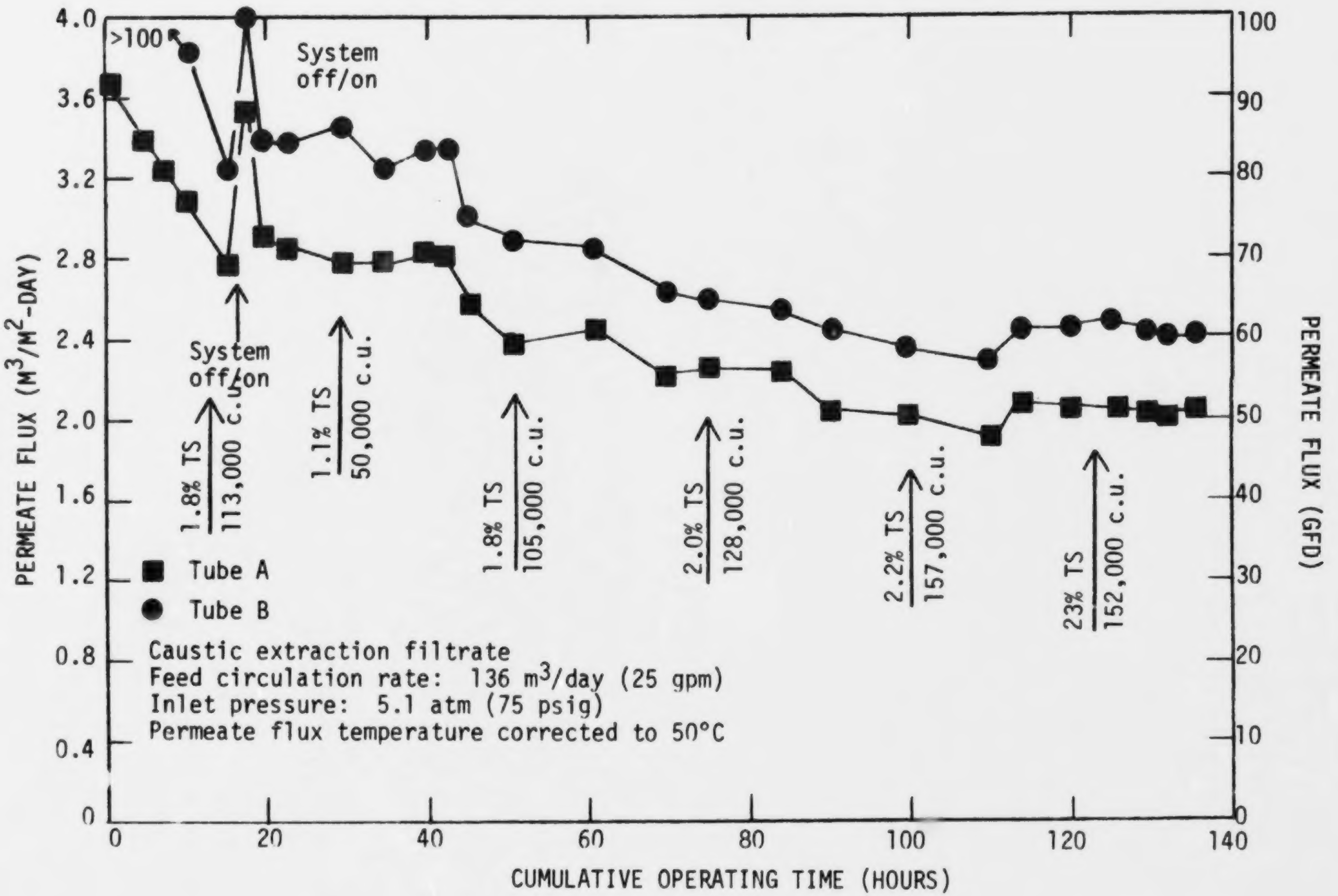


Figure 43. 25.4 mm diameter tubular polysulfone membrane flux versus time during 90% conversion test period.

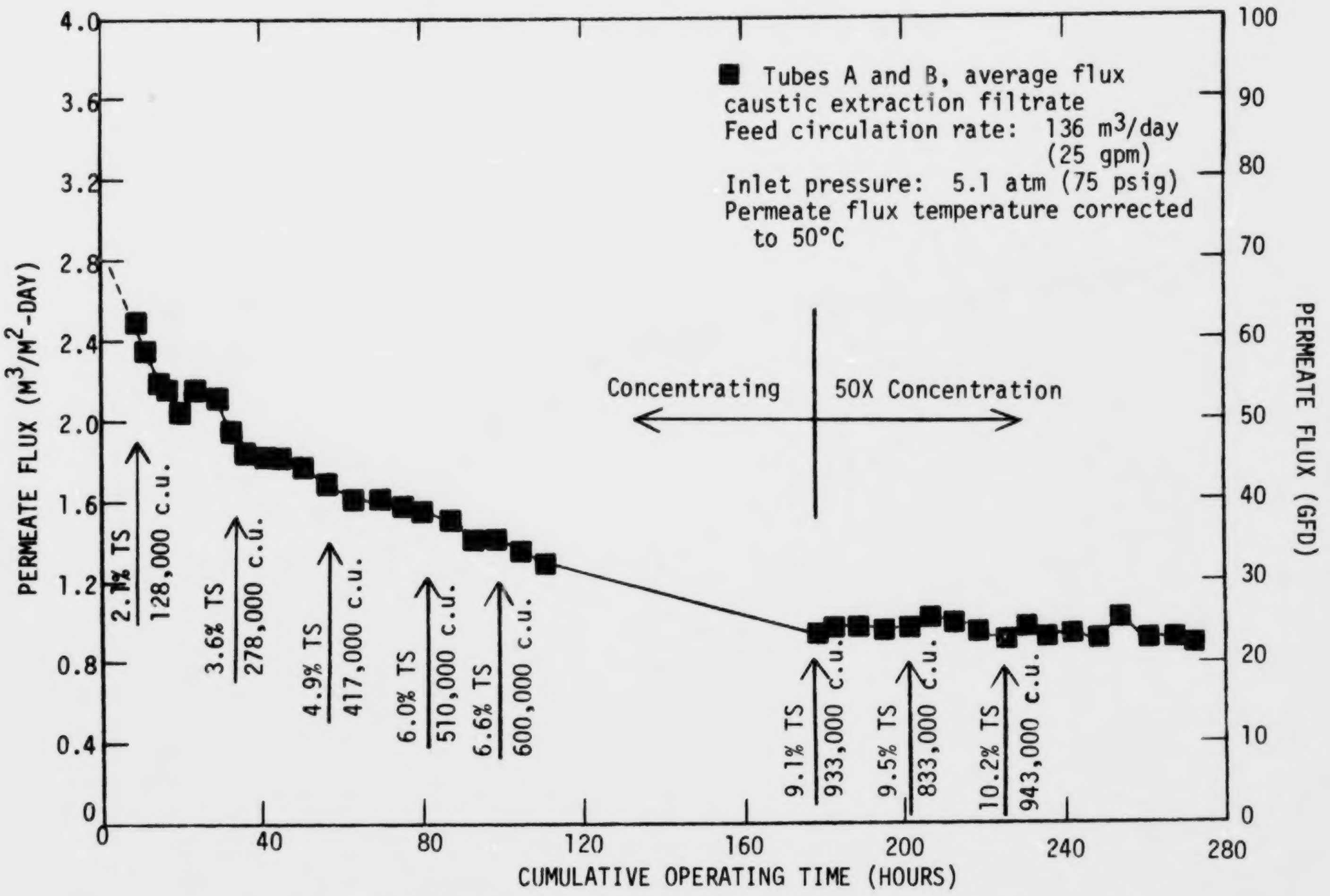


Figure 44. 25.4 mm diameter tubular polysulfone membrane flux versus time during 98% conversion test period.

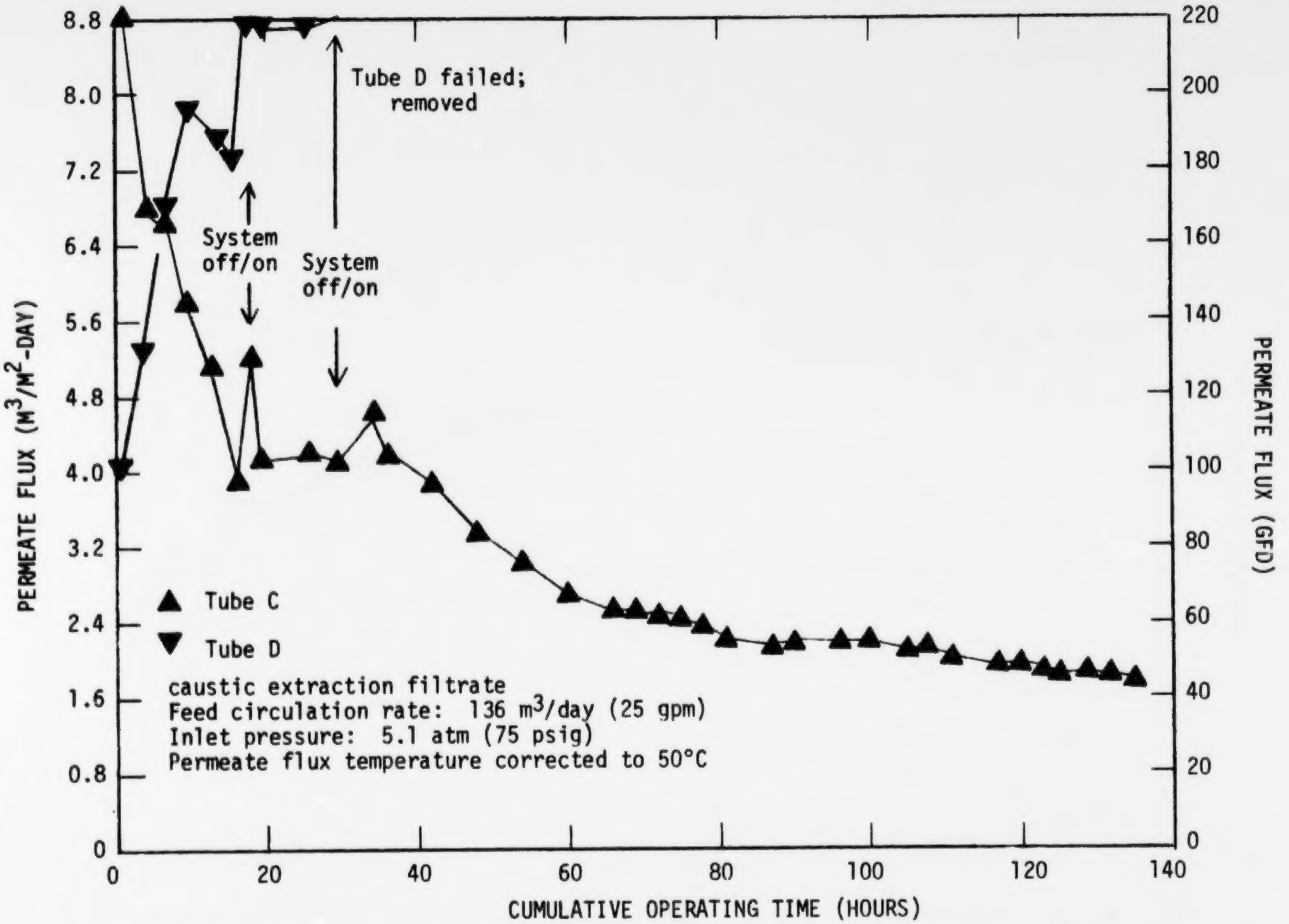


Figure 45. 25.4 mm diameter tubular HFM membrane flux versus time during 90% conversion test period.

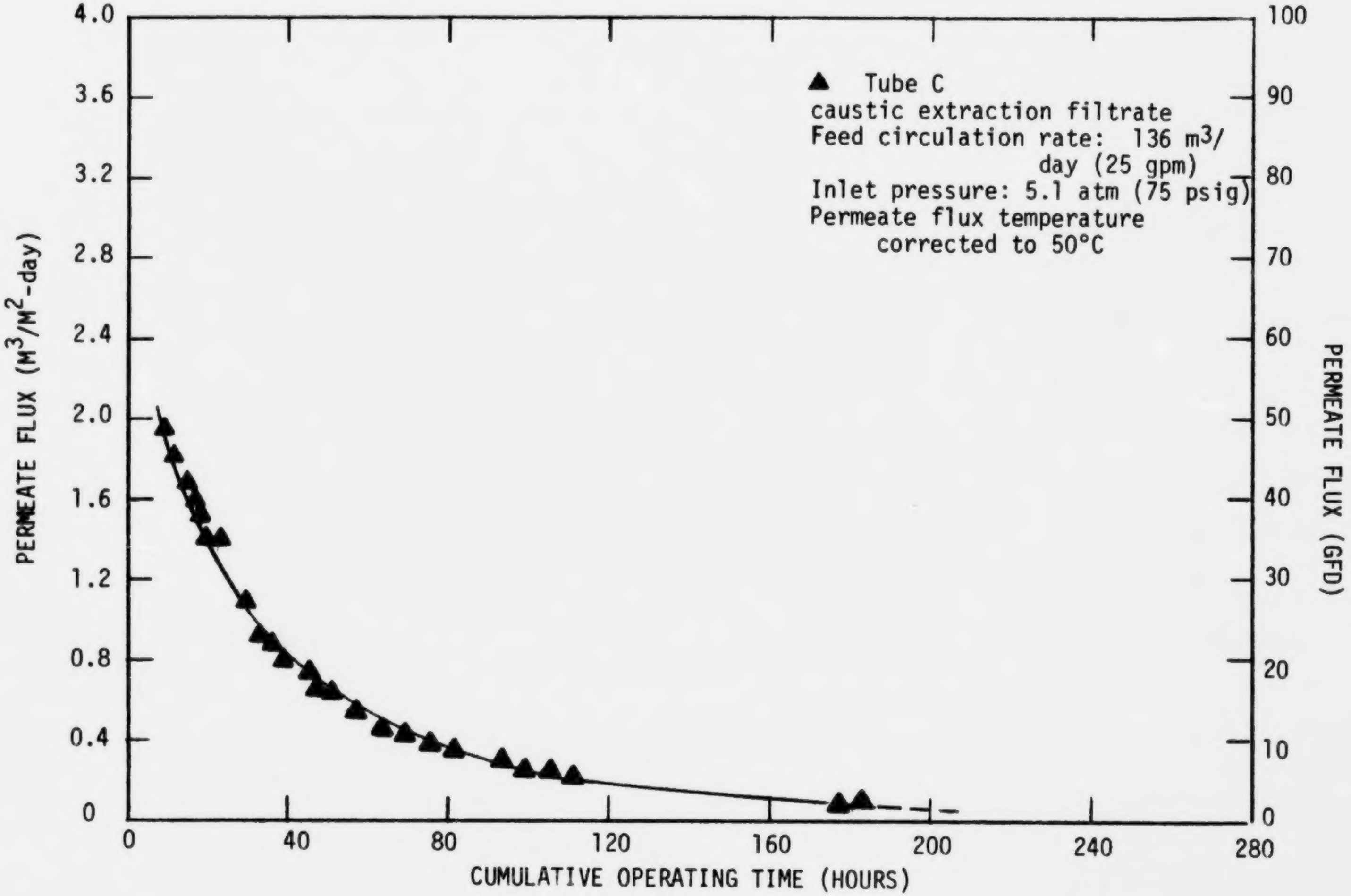


Figure 46. 25.4 mm diameter tubular HFM membrane flux versus time during 98% conversion test period.

TABLE 13. FLUX RECOVERY FOR 25.4 MM DIAMETER TUBULAR POLYSULFONE MEMBRANES

Cumulative operating time, hrs	Inlet pressure, atm (psig)	Water flux @ 50°C, m^3/m^2-day (gfd)		Cleaning conditions
		Tube A	Tube B	
0	6.1 (75)	5.33 (130)	5.86 (143)	New membranes.
133	6.1 (75)	5.21 (127)	6.72 (164)	2-hour ultraclean wash.
268	6.1 (75)	5.45 (133)	7.05 (172)	2-hour ultraclean wash.
541	3.4 (50)	1.76 (43)	2.38 (58)	Membranes stored for 3-months without washing. Then washed for 1-hour with ultraclean/caustic. Partial membrane drying suspected. Note: lower operating pressure.

ultraclean/caustic wash. Resultant water fluxes were 1.76 m^3/m^2-day (43 gfd) for tube A and 2.38 m^3/m^2-day (58 gfd) for tube B. However, these data were recorded at only 3.4 atm (50 psig) operating pressure. Also, partial membrane dry-out is suspected.

HFM tubular membrane flux averaged 24.6 m^3/m^2-day (600 gfd) initially. Subsequent to the 10X concentration run tube C flux was recovered to only 47.6 m^3/m^2-day (116 gfd) with ultraclean. This membrane assembly was not cleaned after the Canton Mill tests. Tube D failed during the 10X concentration period.

Color Rejection--The polysulfone membranes in 25.4 mm (1 in) diameter tubular assemblies produced permeate of acceptable quality at all three system conversions tested. Permeate color concentration and membrane color rejection during these tests are shown in Figures 47 and 48. In the 1.2X concentration (16.7% conversion) runs the average permeate color concentration (both tubes A and B) was 709 color units (c.u.). Throughout this test the feed averaged 20,200 c.u. and the concentrate averaged 25,400 c.u. On a feed basis, average color rejection was 96.5% to 97%. This range of rejection was consistently maintained throughout the run as Figure 47 indicates.

The 10X (90% conversion) period data are also graphed in Figure 47. Average permeate quality after reaching a 10X concentration was 1,870 c.u. Membrane color rejection (feed basis) was consistently 88% to 90%. During this test the average color concentrations were 19,100 for the feed stream and 117,000 for the concentrate.

The 50X (98% conversion) data are plotted in Figure 48. Permeate quality degrades as the concentration with the membrane loop builds up to 50X. After reaching 50X permeate quality averages 10,000 c.u. Mean feed color concentration was 23,400 throughout the test. Concentrate color, after reaching 50X, averaged 927,000. While the membranes rejected 98% to 99% of the color they were exposed to (concentrate basis), actual color rejection on a feed basis ranged from only 50% to 60%.

Projected Color Discharge: Mass Basis--Using the data developed during these tests, projections can be made of color discharge on a mass basis (kilograms of color per day) from each stage of a full-scale system. In developing these projections the following data (and assumptions) are used:

- The UF system consists of 3 stages. Conversions per stage are 16.7%, 90% and 98%, respectively.
- 3,790 m^3/day (1,000,000 gpd) of caustic extraction filtrate are processed.
- Average feed color concentration is 20,000 color units.
- Average permeate color concentrations are:

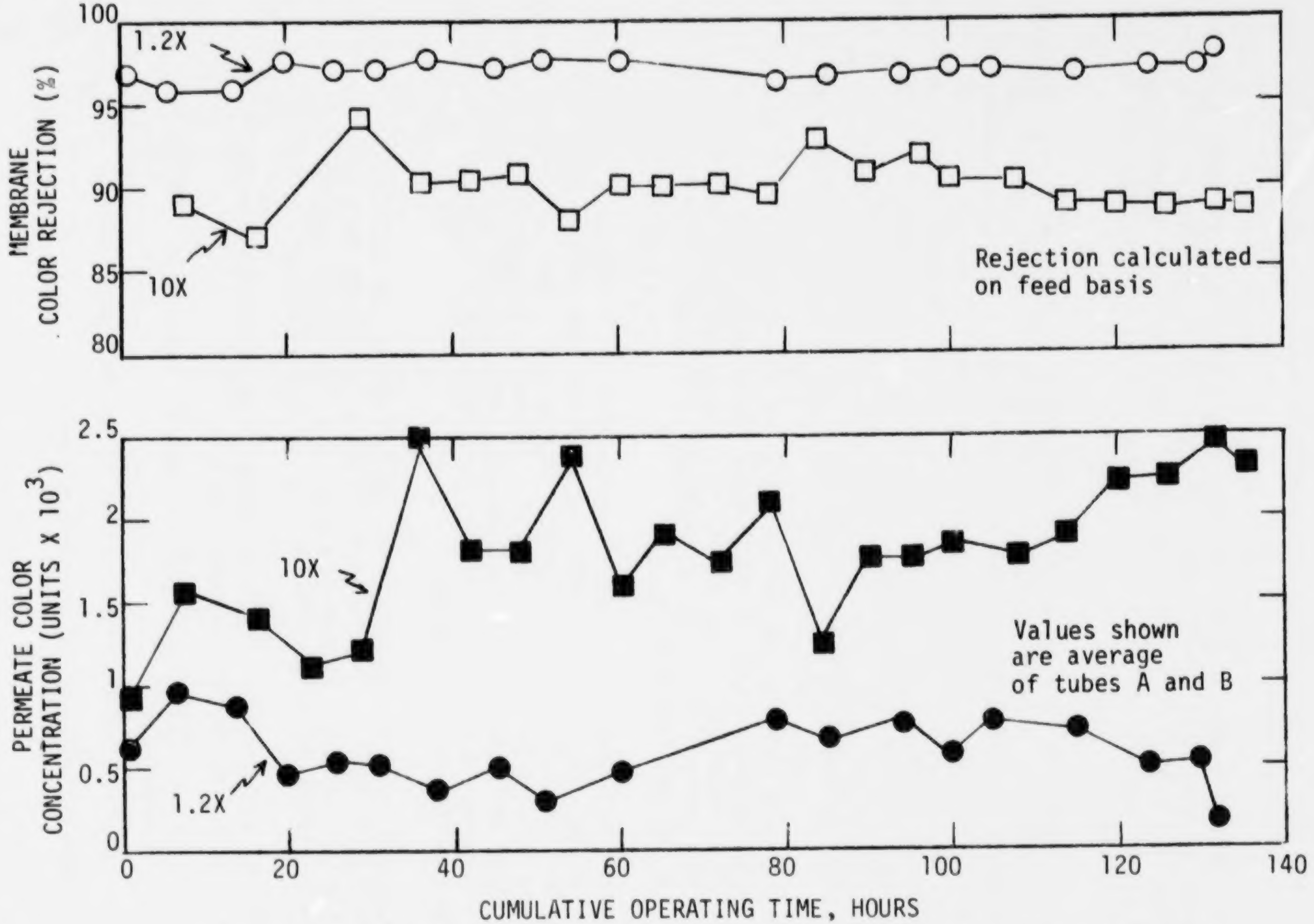


Figure 47. Tubular polysulfone membrane permeate quality and color rejection during 1.2X and 10X concentration periods (caustic extraction filtrate feed).

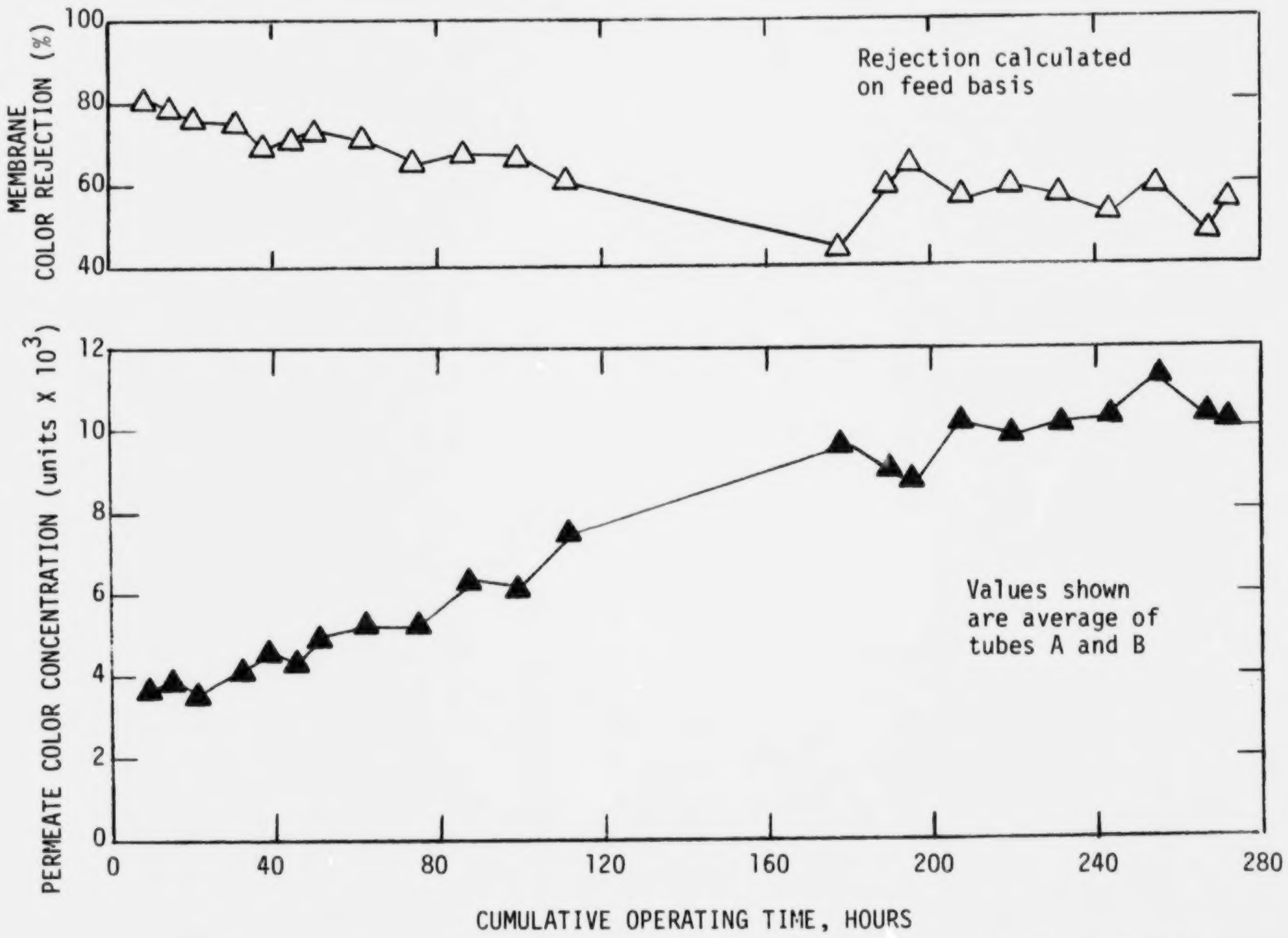


Figure 48. Tubular polysulfone membrane permeate quality and color rejection during 50X concentration period (caustic extraction filtrate feed).

Stage	Color Concentration (c.u.)
1	800
2	2,000
3	10,000

- 8 color units are equivalent to 1 mg/l color discharged.

The stage 1 discharge of color is thus:

$$\begin{array}{l|l|l|l|l} 670{,}000\text{ gal} & 3.785\text{ l} & 800\text{ c.u.} & \text{mg} & \text{kg} \\ \hline \text{day} & \text{gal} & & 8\text{ c.u.-l} & \text{mg} \times 10^6 \end{array} = 253.6\text{ kg/day}$$

Stage 2 color discharge is:

$$\begin{array}{l|l|l|l|l} 230{,}000\text{ gal} & 3.785\text{ l} & 2{,}000\text{ c.u.} & \text{mg} & \text{kg} \\ \hline \text{day} & \text{gal} & & 8\text{ c.u.-l} & \text{mg} \times 10^6 \end{array} = 217.6\text{ kg/day}$$

And for Stage 3:

$$\begin{array}{l|l|l|l|l} 80{,}000\text{ gal} & 3.785\text{ l} & 10{,}000\text{ c.u.} & \text{mg} & \text{kg} \\ \hline \text{day} & \text{gal} & & 8\text{ c.u.-l} & \text{mg} \times 10^6 \end{array} = 378.5\text{ kg/day}$$

Using a similar calculation, if no treatment were employed for color removal 9,462.5 kg/day (20,365 lb/day) of color bodies would be discharged. The overall color reduction in the caustic extraction filtrate stream by employing tubular polysulfone membranes under the assumed conditions and discharging all the permeate to sewer would therefore be:

$$\frac{9{,}462.5\text{ kg}-(253.6\text{ kg} + 217.6\text{ kg} + 378.5\text{ kg})}{9{,}462.5\text{ kg}} = 91.0\%$$

On the assumption that pine bleachery caustic extraction filtrate contributes 60% of the Canton Mill effluent color then the removal of 91% of this color should result in a decrease of 54.6% (0.91 x 0.60 x 100) in the total mill effluent color.

CLEANING EFFECTIVENESS

The cleaning of the spiral wound WRP membrane modules was optimized on the single module test stand. The ease of cleaning was a direct function of how badly fouled the membrane module was before cleaning. If the module was cleaned before 50% flux decline had occurred, the cleaning was easier. However, if the membrane was fouled such that flux had declined to less than 25% of the clean membrane value, the necessary cleaning sequence and time were unpredictable.

The typical WRP membrane spiral wound modules used for this experimental work had initial water flux values at 21°C of 3.28 m^3/m^2-day (80 gfd). After use and cleaning a "clean" membrane had flux values between 1.64 to 2.05 m^3/m^2-day (40 and 50 gfd). This value was reproducible over the number of cleaning cycles done on the various membrane modules. The individual modules had insufficient time on stream to predict membrane life or long term cleanability.

Flux decline and fouling conditions were not observed with the polysulfone tubular membranes. Between experimental runs (typically one week in duration) the tubes were cleaned with the cleaning solution found optimum for WRP spiral wound modules. This cleaning recovered water flux values equivalent to the initial values.

The flux decline, fouling problems and cleaning problems encountered with the WRP spiral wound modules were not found with tubular membranes. While no problems were observed and none are predicted from the operating experience, long term cleanability data are not available for tubular membranes operated on pulp mill and bleachery effluents.

MATERIAL BALANCE

In the course of the four week pilot run with 25.4 mm diameter tubular membranes, samples were collected for material balance analyses. Samples of feed, permeate and concentrate were collected for each assembly at concentration ratios of 1.2X, 10X and 50X. Color and total solids analyses were conducted on the as-collected samples in the Champion Laboratory. Composited samples were analyzed chemically at Galbraith Laboratories, Knoxville, Tenn. These analyses are presented in Appendix G.

A flow schematic for identification of the samples collected from this system is presented in Figure 49. A material balance for this system consists of the comparison of the mass flow and constituent composition of the feed material - in this case, pine bleachery caustic extraction filtrate - to the sum of the mass flows and constituent compositions of the final concentrate and the composited permeates from each stage.

There are a number of non-controllable parameters in operating a pilot system on a "live" operating plant effluent. For example, the feed composition can vary on an hour to hour and week to week basis in an unpredictable manner. In addition, the substrate for analysis is a very complex mixture of organic and inorganic dissolved, nuclear and agglomerated materials which renders precise chemical analysis difficult. In addition to these considerations, for pragmatic purposes, it was necessary to reduce the total number of samples for analysis by preparing composites of a number of like samples collected over a period of time. The material balance obtained under these constraints is valuable in providing a set of guidelines and an understanding of the system but it is not interpretable as a precise delineation of the system in all respects.

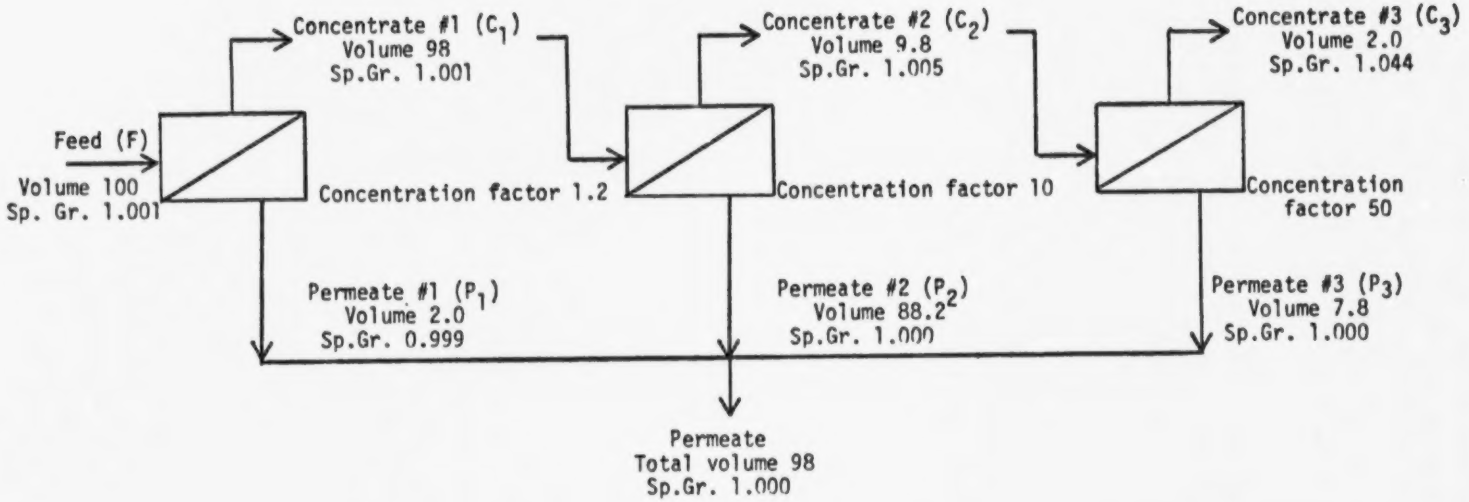

Concentrate #1 (C_1)
Volume 98
Sp.Gr. 1.001
Concentrate #2 (C_2)
Volume 9.8
Sp.Gr. 1.005
Concentrate #3 (C_3)
Volume 2.0
Sp.Gr. 1.044
Feed (F)
Volume 100
Sp. Gr. 1.001
Concentration factor 1.2
Concentration factor 10
Concentration factor 50
Permeate #1 (P_1)
Volume 2.0
Sp.Gr. 0.999
Permeate #2 (P_2)
Volume 88.2
Sp.Gr. 1.000
Permeate #3 (P_3)
Volume 7.8
Sp.Gr. 1.000
Permeate
Total volume 98
Sp.Gr. 1.000

Figure 49. Flow schematic for identification of samples from material balance studies with caustic extraction filtrate (25.4 mm diameter tubular polysulfone membranes).

Relative Material Balance

A relative material balance for the system during concentration to a 50X concentration ratio is presented in Figure 50. In this figure each component is considered as 100% in feed concentration. The amount of the component relative to the feed concentration is presented for the final concentrate and the composited total permeate from the system. As may be noted, the color constituents are retained in the concentrate (92.6% of the feed). The majority of the total solids are contained in the permeate (64.8%) as might be expected due to the large amount of ionic and small molecular weight organic materials in this feed stream. All metal ions show some rejection over the membrane varying from rejections of 100% for aluminum to a rejection of 7.6% for sodium.

The sulfate and chloride ion distributions can be interpreted to indicate that these species are expedited in passage through the membrane because they are identified at low levels in the concentrate. This is discussed in more detail below. It is of interest to note that the organic chloride is predominately in the permeate. This indicates that a large amount of this chlorine is associated with low molecular weight materials like chloroform which pass through the membrane easily.

A graphical display of the detailed chemical analyses by ultrafiltration stage is presented in Figure 51. This figure provides information on the measured values of various constituents. Examination of this data will provide information which would be too voluminous to include here. For example, from the C_3 concentrate data one may obtain a rough equivalence figure for conversion of color as measured in color units to actual parts per million by weight content; e.g., dividing the color (927,000 c.u.) by the solids content (102,800 ppm) indicates that roughly 9 color units are equivalent to 1 ppm solution solids by weight.

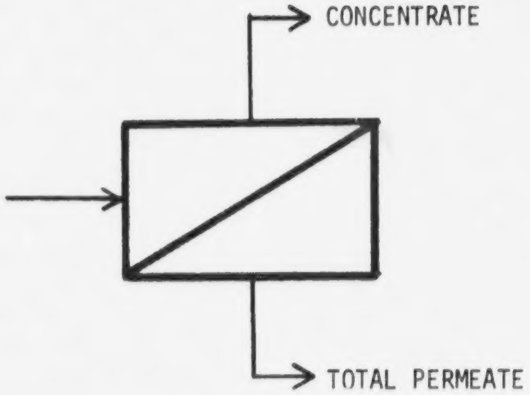
Ion Rejection by the Membrane

The concentration of various elemental materials in the ultrafiltration concentrates obtained by operating at several concentration ratios is presented in Figure 52. The lower chart presents data for aluminum, calcium, iron and sodium. The ion concentration ratio is the ion content in the concentrate relative to that in the feed. If a metal were rejected 100% by the membrane, then at a 50X concentration ratio the ion concentration ratio should be 50X. As can be observed this is the case for aluminum. Iron is rejected about 92%. Calcium is rejected about 65% and sodium is rejected about 7.6%.

These rejections may be related to charges on the species and a like electrostatic charge on the membrane surface. The rejection may also be due to chemical complex formation by the multivalent ions with other materials in the system. Irrespective of the explanatory mechanism, this metal behavior has economic importance. The removal of the metals improves the quality of the permeate and allows for broader utility of this stream for

FEED

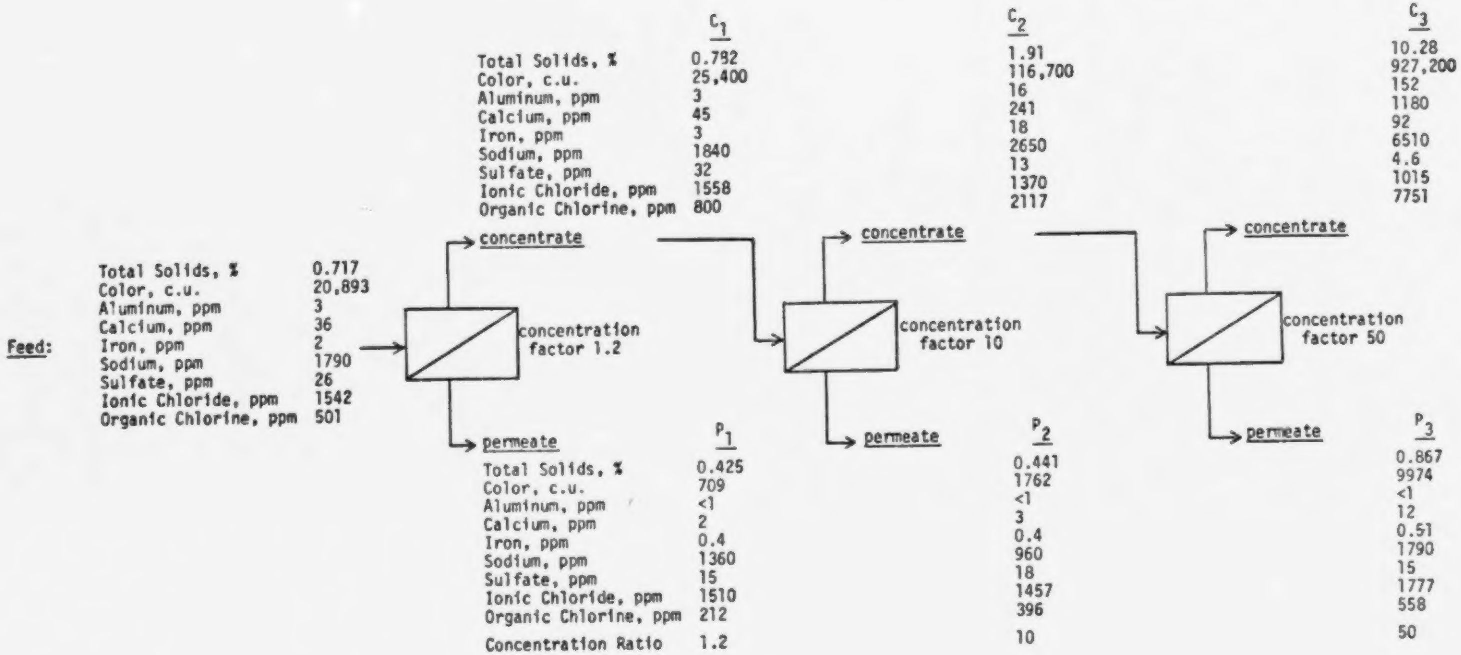
	%
Water	100
Color	100
Total Solids	100
Aluminum	100
Calcium	100
Iron	100
Sodium	100
Sulfate	100
Ionic Chloride	100
Organic Chloride	100



	%
Water	2
Color	92.6
Total Solids	29.9
Aluminum	100
Calcium	68.4
Iron	96
Sodium	7.6
Sulfate	0.4
Ionic Chloride	1.4
Organic Chloride	32.3

	%
Water	98
Color	11.2
Total Solids	64.8
Aluminum	--
Calcium	10.1
Iron	20
Sodium	56.6
Sulfate	66.6
Ionic Chloride	94.2
Organic Chloride	79.2

Figure 50. Relative material balance for 50 times concentration on pine bleachery caustic extraction filtrate using 25.4 mm diameter tubular polysulfone membranes.


concentrate
concentration factor 1.2
permeate
concentrate
concentration factor 10
permeate
concentrate
concentration factor 50
permeate

Feed:

Total Solids, %	0.717
Color, c.u.	20,893
Aluminum, ppm	3
Calcium, ppm	36
Iron, ppm	2
Sodium, ppm	1790
Sulfate, ppm	26
Ionic Chloride, ppm	1542
Organic Chlorine, ppm	501

	C_1	C_2	C_3
Total Solids, %	0.792	1.91	10.28
Color, c.u.	25,400	116,700	927,200
Aluminum, ppm	3	16	152
Calcium, ppm	45	241	1180
Iron, ppm	3	18	92
Sodium, ppm	1840	2650	6510
Sulfate, ppm	32	13	4.6
Ionic Chloride, ppm	1558	1370	1015
Organic Chlorine, ppm	800	2117	7751

	P_1	P_2	P_3
Total Solids, %	0.425	0.441	0.867
Color, c.u.	709	1762	9974
Aluminum, ppm	<1	<1	<1
Calcium, ppm	2	3	12
Iron, ppm	0.4	0.4	0.51
Sodium, ppm	1360	960	1790
Sulfate, ppm	15	18	15
Ionic Chloride, ppm	1510	1457	1777
Organic Chlorine, ppm	212	396	558
Concentration Ratio	1.2	10	50

Figure 51. Analyses of pine caustic extraction filtrate at various stages of ultrafiltration using 25.4 mm diameter polysulfone tubular membranes.

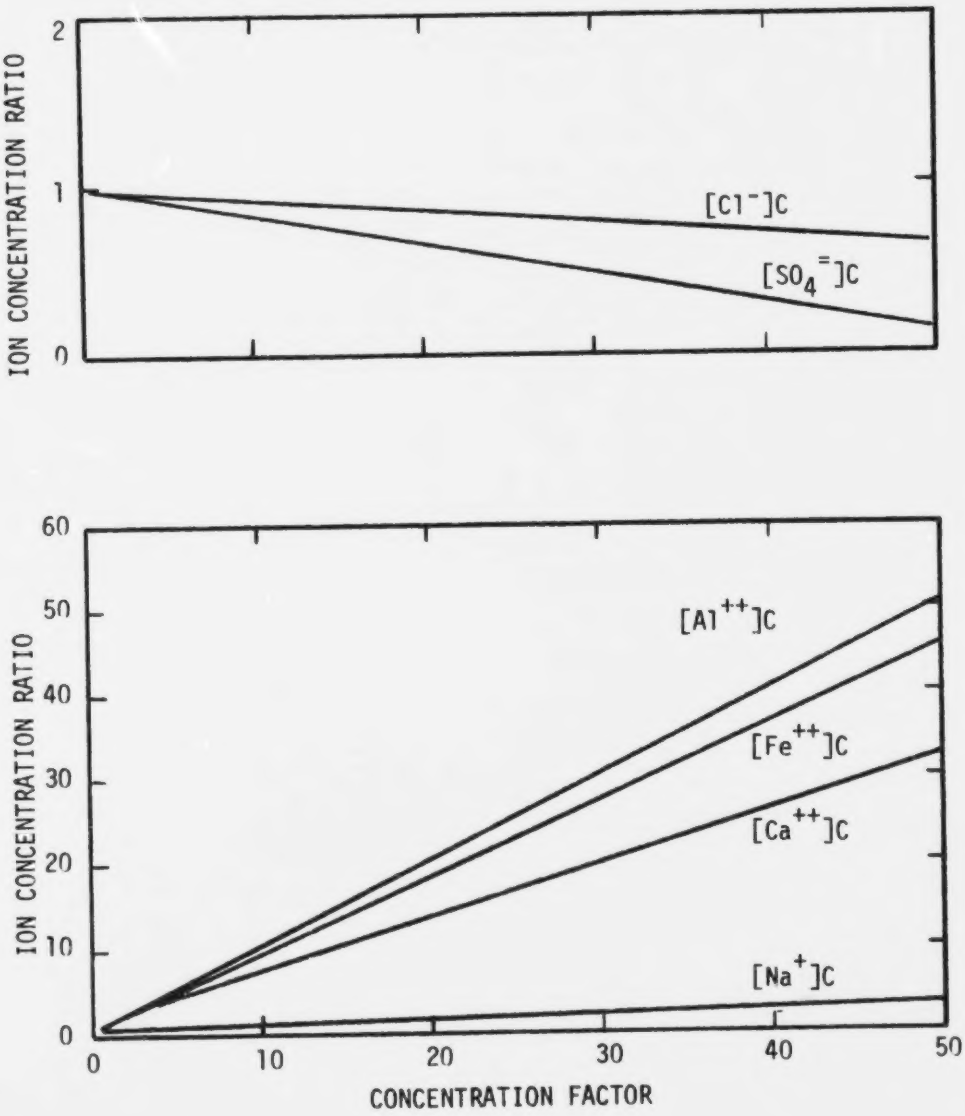


Figure 52. Ion concentration versus concentration ratio for ultrafiltration of caustic extraction filtrate (25.4 mm diameter tubular polysulfone membranes).

recycle. The low rejection of sodium is important in ultrafiltration. Reducing the sodium level minimizes the osmotic pressure build-up problems on concentration and consequently allows for operation of ultrafiltration at high concentration levels and relatively low system pressure.

The upper chart in Figure 52 presents ion concentration data for sulfate and chloride ion as a function of concentration factor. As presented, it appears that these ions are removed from the concentrate as the concentration factor is increased and also that the sulfate (with the higher charge) is expedited out of the concentrate more readily than the chloride. The sulfate appears to be about 20% of what would be anticipated and the chloride about 67%.

This phenomenon is not explainable with the data at hand. There are, however, a number of possible explanations. First, in this complex system it is possible that the analytical techniques employed are not adequate for the purpose. Secondly, it is possible that in the concentrated material the sulfate ion especially might be tied into a complex with other materials and hence effectively removed from the analytical sample. A third possible explanation may be posited on the basis of membrane surface charges and their influences in expediting passage of anions into the permeate.

Organic Chlorine

Analyses were made on each sample for ionic chloride, total chlorine and also for volatile materials. The difference between the value for a sample of total chlorine and ionic chloride is assumed to be chlorine bonded to organic material. The volatile content of the sample solids is assumed to be a measure of the total organic content. The ratio of the non-ionic chloride to the volatile content is then a measure of the chlorine content of the organic materials. In previous work it has been demonstrated that for pine bleachery caustic extraction filtrate color bodies the chlorine content is about 8%.

In Figure 53 the calculated ratios of non-ionic chlorine to volatiles (% chlorine content of the organic materials) is presented for the permeates and concentrates as a function of concentration factor.

The organic chlorine content of the materials in the permeate appear to rise rapidly in the initial stages of ultrafiltration concentration and then decrease as concentration is continued. The organic chlorine content of the material in the concentrate monatonically decreases with concentration and at 50 times concentration are approaching the anticipated value.

Removal of the low molecular weight chlorinated materials - especially materials like chloroform - is felt to be the basis for the results obtained. In the feed substrate there are a number of chlorinated materials possible which should be capable of free passage through the membrane, all with organic chlorine contents which are high (e.g. chloroform at 89.1%) compared to the

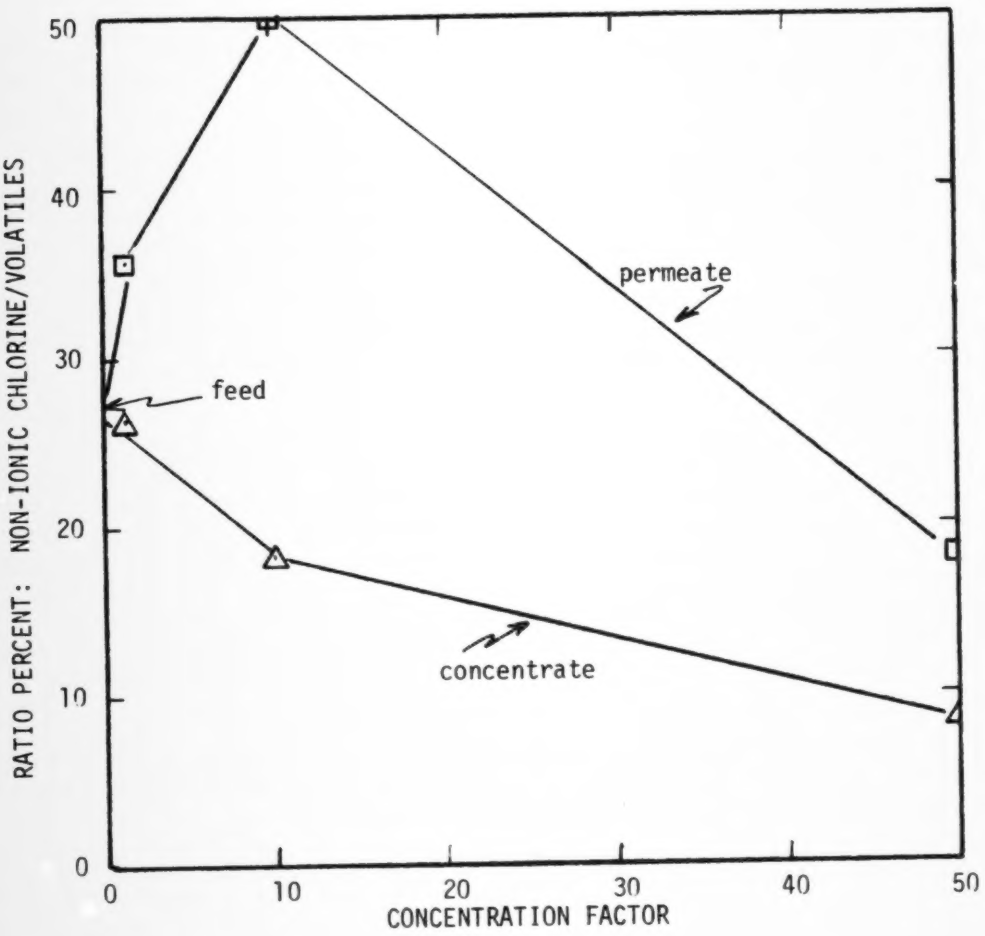


Figure 53. Ratio of non-ionic-chlorine/volatiles versus concentration factor for ultrafiltration of caustic extraction filtrate (25.4 mm tubular polysulfone membranes).

color bodies (about 8% chlorine). As these small, high chlorine content materials are removed in the permeate, the type of results obtained would be expected.

Specific Gravity

In the course of the analyses the specific gravity for each of the samples was measured. These results are presented in Figure 54. As presented, the specific gravity of the permeates to 50 times concentration is constant at 1.000. The specific gravity of the concentrate increases with increasing concentration.

The inherent density of the solids in the concentrate have been calculated from this data and appear to be about 1.43 gms/ml.

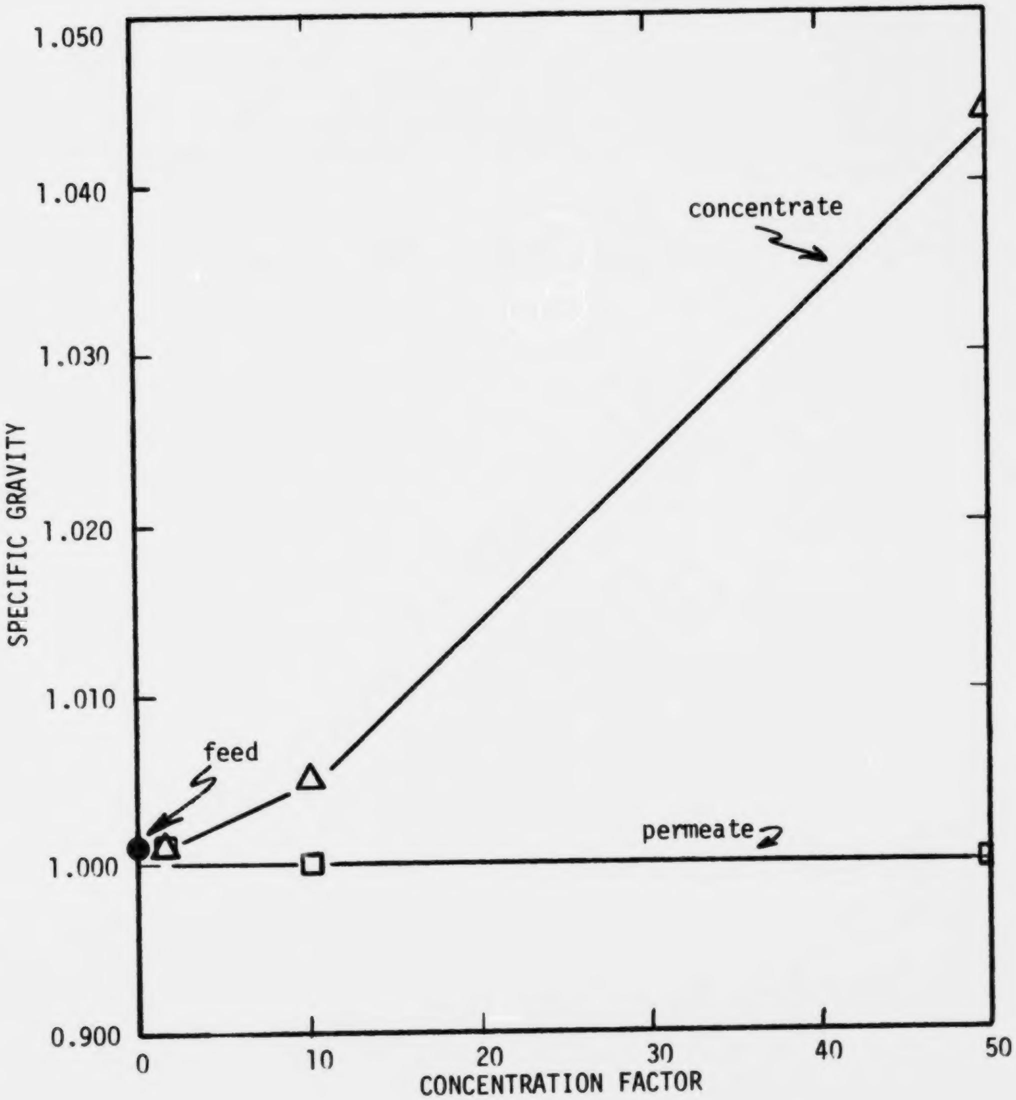


Figure 54. Specific gravity versus concentration factor for ultrafiltration of caustic extraction filtrate (25.4 mm diameter tubular polysulfone membranes).

SECTION 7

CONCEPTUAL DESIGN

INTRODUCTION

Eight design cases have been reviewed in developing economic projections for ultrafiltration of kraft pulp mill effluents. These 8 cases, summarized in Table 14, encompass the 3 streams of interest: caustic extraction filtrate, pine decker and hardwood decker; two module geometries: tubular and spiral-wound; and, two system capacities: 3,790 and 7,580 m^3/day (1 MM and 2 MM gpd). The 3,790 or 7,580 m^3/day (1 or 2 MM gpd) caustic extraction filtrate would come from bleaching 727 metric tons/day (800 tons/day) of pine pulp. The 3,790 or 7,580 m^3/day (1 or 2 MM gpd) decker effluent would come from washing 1,318 metric tons/day (1450 tons/day) of mixed pine and hardwood pulp. The system of most potential interest, a 3,790 m^3/day (1 MM gpd) tubular system treating caustic extraction filtrate (Case 1), will be discussed in detail. The remaining seven cases will be discussed as variants of the Case 1 design.

The flux levels used for design of the tubular systems were derived from the experimental data. The same flux levels were used in designing the spiral wound systems in the anticipation of the development of improved module spacer designs.

DETAILS OF CASE 1 DESIGN

Summary

The Case 1 design bases are as follows:

- -- 3,790 m^3/day (1×10^6 gpd) pine caustic extraction filtrate processed.
- -- tubular membrane assemblies used. Each assembly 25.4 mm (1 in) diameter x 3.05 m (10 ft) long, interconnected to form a 6.1 m (20 ft) length. Each parallel pass of membranes containing 8 6.1 m (20 ft) lengths of membrane connected in series with U-bends. Total membrane area per parallel pass is 3.27 m^2 (35.2 ft^2).
- -- prefiltration consists of a hydrasieve for fiber removal and a backflushable 5-10 μ sock filter.

TABLE 14. FULL-SCALE SYSTEM DESIGN CASES

Case number	Feed stream	Flowrate, m^3/day (MM gpd)	Quantity of pulp produced, metric tons/day (tons/day)	Module type	Overall system conversion
1	Caustic extraction filtrate	3,790 (1)	727 (800)	tubular	98%
2	Caustic extraction filtrate	7,580 (2)	727 (800)	tubular	98%
3	Pine and hardwood decker	3,790 (1)	1,318 (1,450)	tubular	95%
4	Pine and hardwood decker	7,580 (2)	1,318 (1,450)	tubular	95%
5	Caustic extraction filtrate	3,790 (1)	727 (800)	spiral-wound	98%
6	Caustic extraction filtrate	7,580 (2)	727 (800)	spiral-wound	98%
7	Pine and hardwood decker	3,790 (1)	1,318 (1,450)	spiral-wound	95%
8	Pine and hardwood decker	7,580 (2)	1,318 (1,450)	spiral-wound	95%

-- the ultrafiltration system consists of 3 stages. These stages, detailed in Table 15, contain a total of seven subsystems.

-- a 10% excess has been calculated into the membrane area requirements as a safety factor.

-- a single cleaning station is included with the UF system. This station is capable of automatic cleaning and rinsing of any one of the subsystems at any time.

-- the UF permeate is collected in a holding tank (1 hour residence). From here it is recycled within the mill, used for system cleaning operations or sewered.

-- the UF concentrate is returned to the weak black liquor, to the lime kiln or to landfill, or mixed with lime sludge.

Flow Schematic Description

A flow schematic of the Case 1 system is shown in Figure 55. A flow of 3,790 m^3/day (700 gpm) of first stage pine caustic extraction filtrate is continuously fed into a hydrasieve for fiber removal. While 3,790 m^3/day (700 gpm) is the average feed flow rate, it is expected that the system can handle reasonable flow fluctuations (±10%) with little, if any, difficulty. Should a serious mechanical problem occur downstream of the hydrasieve, a bypass to sewer is provided. Also provided is a port for permeate feed to the hydrasieve to maintain the entire system in recycle operation should the caustic line go down.

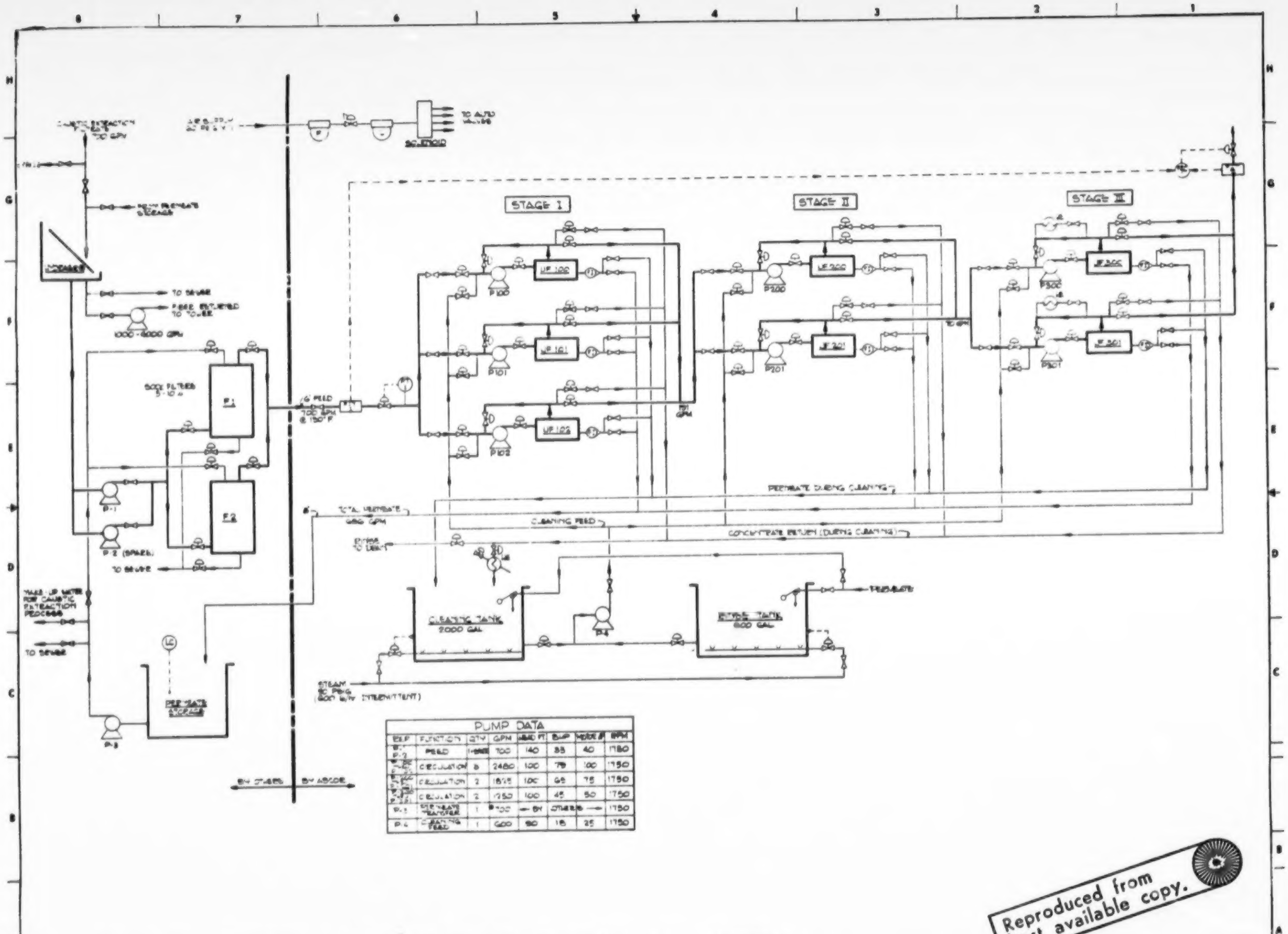
Fiber in the feed stream will be removed by the hydrasieve and recycled to the tower. If necessary, the fiber-laden stream can be sewered. The hydrasieve screening will have a flowrate of 91 to 364 m^3/day (1,000 to 4,000 gph).

The underflow from the hydrasieve will be passed through a backwashable finger filter by a 3,790 m^3/day (4.1 atm head, 1750 RPM) [700 gpm (60 psi head, 1750 RPM)] centrifugal pump equipped with a 27.8 kw (40 hp) motor. A spare 3,790 m^3/day (700 gpm) pump is provided to maintain system operability during maintenance periods. The finger filter consists of cylindrical stainless steel screens outfitted with polypropylene "socks". Solids on the order of 5 to 10 μ will be retained by the filters which are automatically backflushed with UF permeate when a critical pressure differential between the inlet and outlet headers is reached. Two 3,790 m^3/day (700 gpm) filter systems are provided; a clean system being on standby at all times.

The effluent from the sock filters flows into the first stage of the UF system. This stage is made up of 3 identical subsystems each containing 327 m^2 (3,520 ft^2) of membrane area. A conceptual design of a subsystem is shown in Figure 56. In its simplest terms a subsystem consists of membranes, a membrane support rack and a pumping station.

TABLE 15. ULTRAFILTRATION SECTION DESIGN -- CASE 1

Item	Stage number 1	2	3
Total membrane area, m^2 (ft^2)	981 (10,560)	477.5 (5,140)	327 (3,520)
No. of subsystems	3	2	2
Membrane area per subsystem, m^2 (ft^2)	327 (3,520)	238.8 (2,570)	163.5 (1,760)
No. of parallel membrane passes per subsystem	100	73	50
Circulation flowrate per subsystem, m^3/day (gpm)	13,516 (2,480)	9,946 (1,825)	6,813 (1,250)
Pressure drop per parallel pass, atm (psi)	2.38 (35)	2.38 (35)	2.38 (35)
Motor power, kW (hp)	74.6 (100)	55.9 (75)	37.3 (50)
Efficiency, %	80	74	70
Brake power per subsystem, kW (hp)	58.9 (79)	47.0 (63)	33.6 (45)
Design flux, m^3/m^2-day (gfd)	2.87 (70)	2.05 (50)	1.03 (25)
Typical conversion, feed basis, %	67	90	98


STAGE I
STAGE II
STAGE III
UF 100
UF 101
UF 102
UF 200
UF 201
UF 300
UF 301
P100
P101
P102
P200
P201
P300
P301
CAUSTIC EXTRACTION FILTRATE 700 GPM
AIR SUPPLY
SOLENOID
TO AUTO VALVES
TO SEWER
FIBER RETURNED TO TOWER
1000-6000 GPM
SOCK FILTERS 5-10 μ
F1
F2
P-1
P-2 (SPARE)
P-3
P-4
6" FEED 700 GPM @ 150°F
TOTAL PERMEATE 680 GPM
PERMEATE DURING CLEANING
CLEANING FEED
CONCENTRATE RETURN (DURING CLEANING)
CLEANING TANK 2000 GAL
RINSE TANK 600 GAL
PERMEATE
STEAM 50 PSIG (600 lb/hr INTERMITTENT)
MAKE-UP WATER FOR CAUSTIC EXTRACTION PROCESS
TO SEWER
LC
PERMEATE STORAGE
BY OTHERS
BY ABCOR

PUMP DATA

REF	FUNCTION	QTY	GPM	HEAD FT	BHP	MOTOR HP	RPM
P-1 P-2	FEED	1+[illegible]	700	140	83	40	1780
P-100 P-101 P-102	CIRCULATION	3	2480	100	79	100	1750
P-200 P-201	CIRCULATION	2	1825	100	65	75	1750
P-300 P-301	CIRCULATION	2	1250	100	45	50	1750
P-3	PERMEATE TRANSFER	1	700	← BY	OTHERS	→	1750
P-4	CLEANING FEED	1	600	90	18	25	1750

Figure 55. Proposed 3,790 m^3 (1 MM gpd) UF system flow schematic.

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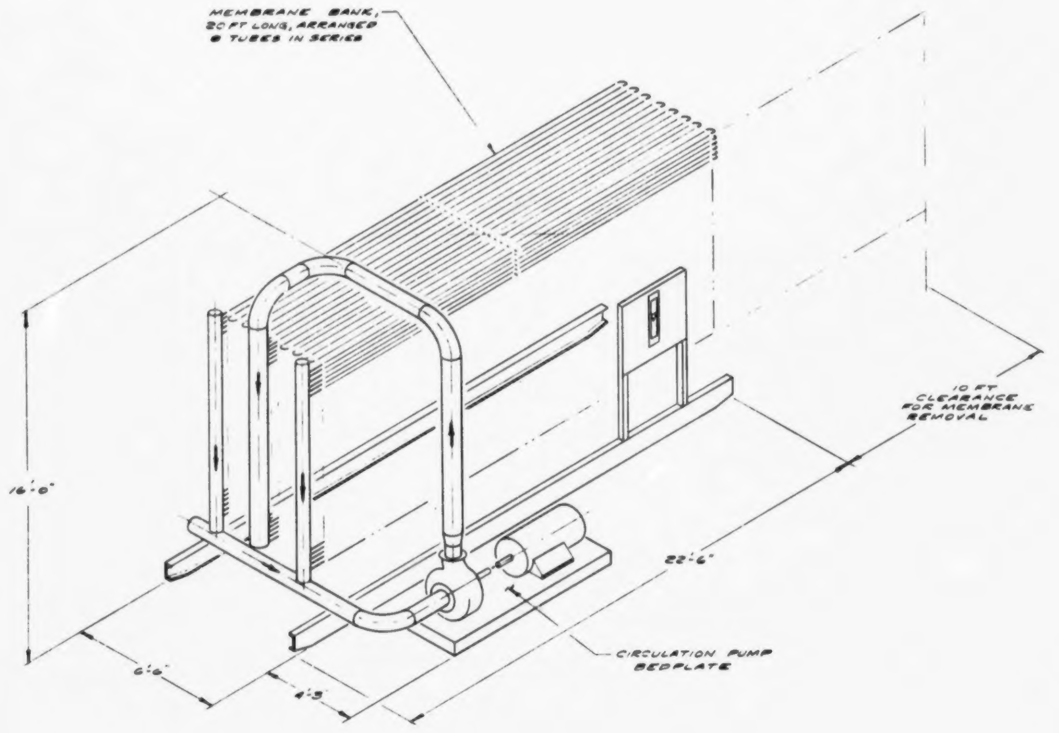
MEMBRANE BANK, 20 FT LONG, ARRANGED 8 TUBES IN SERIES
10 FT CLEARANCE FOR MEMBRANE REMOVAL
16'-0"
6'-6"
4'-3"
22'-6"
CIRCULATION PUMP BEDPLATE

Figure 56. Proposed 3,790 m^3 (1 MM gpd) system typical subsystem outline drawing.

The polysulfone membranes are in a tubular geometry. Two 25.4 mm (1 in) diameter x 3.05 m (10 ft) long (current technology) membrane sections are interconnected to form a 6.1 m (20-ft) membrane length. Threaded U-bends "turn-around" the flow to the next tubular assembly in series. Eight membranes (and 7 U-bends) comprise one parallel pass. The pressure drop across a pass is 2.38 atm (136 m^3/day recirculation, 54°C) [35 psig (25 gpm recirculation, 130°F)]. With a 5.1 atm (75 psig) inlet pressure, the average operating pressure is thus 3.91 atm (57.5 psig).

Each pass of 8 membranes provides 3.27 m^2 (35.2 ft^2) of membrane area. As needed, parallel passes can be added to the membrane rack to provide the desired subsystem membrane area.

The membrane support racks are rigid, steel units which support the weight of the membranes and the fluid being processed. A central distribution header carries the feed stream to the membrane inlets. Two inlet nipples are situated side-by-side enabling one parallel membrane pass to be supported on each side of the central distribution header. A single-nipple header on each side of the rack collects the concentrate and returns it to the suction of the circulation pump. The individual tube permeates are collected at the opposite end of the rack and manifolded. Permeate flow is measured by a flow meter.

A skid-mounted pumping station capable of providing 136 m^3/day (25 gpm) recirculation flow (at 5.1 atm) [75 psig] through each parallel membrane pass is provided. The centrifugal circulation pump is interlocked with a temperature indicator-controller and equipped with high and low pressure shutdown switches with audio and visual alarms.

The concentration bleed from the stage 1 subsystems is combined and fed into the second stage of the ultrafiltration system. Stage 2 consists of 2 subsystems, each containing 239 m^2 (2,570 ft^2) of membrane area (73 parallel passes of membranes). These subsystems are similar in design to the stage 1 subsystem.

In a like manner, the Stage 2 concentrate will flow into the third stage of the system for further concentration. This stage has 2 subsystems, each with 163.5 m2 (1,760 ft^2) of membrane area (50 parallel passes).

Typical conversions across each system stage are:

Stage	Conversion
1	67%
2	90%
3	98%

The final concentrate (from stage 3) is bled off at a controlled rate by a flow ratio controller. This controller receives signals of the feed flowrate and ratios the final concentrate flow to maintain the desired overall system conversion. At a feed flowrate of 3,790 m^3/day (700 gpm), 76.3 m^2/day (14 gpm) of concentrate would be discharged. This flow would be directed to either the lime kiln, to the weak black liquor, or admixed with lime sludge.

The permeate produced by each stage is combined and flows into a 189.5 m^3 (50,000 gal) holding tank. This tank provides a residence time of approximately 70 minutes. The permeate is used for several functions in the operation of the overall treatment system:

-- make-up water for UF system cleaning solutions;

-- rinse water for UF system cleaning;

-- backflush water for sock filters;

-- feed water to maintain system operation in recycle if the caustic extraction filtrate line is down.

These uses, however, require only a fraction of the permeate flow. Some portion of the permeate (5% to 50%) may be recycled to the bleachery. The remainder may be blended with the plant treated water supply. Alternatively, the permeate may be sewered.

Cleaning Sequence

Each of the 7 subsystems is cleaned independently, as needed. A single cleaning station consisting of a 7.58 m^3 (2000 gal) cleaning tank, 2.27 m^3 (600 gal) rinse tank, a cleaning pump and associated solenoid valves, timers and piping is supplied with the ultrafiltration system.

It is anticipated that each subsystem will require one cleaning cycle per two week period. The length of each cleaning cycle will probably vary per stage and range from 1 to 3 hours. The conversion across each stage will "swing" as the individual subsystems are being cleaned. This "swing" will be handled automatically by the flow ratio controller receiving signals from the Stage 1 feed inlet and stage 3 concentrate outlet. During the cleaning "swing" periods the second stage of the UF system is pressed upon to work hardest. For this reason the stage 2 membrane area is ∿50% over that needed if no subsystems were ever shutdown for cleaning. However, even with this increase in membrane area, stage 2 will not be able to handle the infrequent stage 1 subsystem cleaning cycles without a "swing" in system conversion.

Overall, the system will have one subsystem in a cleaning mode less than 4% of the time.

DETAILS OF CASE 2 DESIGN

Summary

The Case 2 design bases are as follows:

- -- 7,580 m^3/day (2 x 10^6 gpd) pine caustic extraction filtrate;
- -- tubular membrane assemblies;
- -- hydrasieve and sock filter prefiltration;
- -- three-stage UF system (see Table 16).

Description

The increase in system capacity from 3,790 to 7,580 m^3/day (1 to 2 MM gpd) has no effect on overall system operation. In fact, the only changes which do occur are in the number of subsystems per stage and the capacity of each subsystem. For the Case 2 design, 10 identical subsystems are employed. Each subsystem contains 327 m^2 (3,520 ft^2) of membrane area (100 parallel passes of 8 6.1 m tubes, in series). There are 6 subsystems in Stage 1, 2 subsystems in Stage 2 and 2 subsystems in Stage 3. As with design Case 1 a 10% excess membrane area is provided as a safety factor. However, unlike Case 1, the Case 2 system does not have the extra "swing" capacity built into Stage 2. The individual stage conversion will still shift as a subsystem is being cleaned, but a loss in overall conversion may occur more frequently. The impact of less membrane area in Stage 2 will, however, be relatively minor since with 6 subsystems in Stage 1, a smaller percentage of membrane area will be off line during any one cleaning operation.

It is important to note that many alternative designs exist for both the 3,790 and 7,580 m^3/day (1 and 2 MM gpd) systems. For example, a 7,580 m^3/day (2 MM gpd) system may preferably consist of 2 identical, independent 3,790 m^3/day (1 MM gpd) systems. In that way, a prolonged shutdown of any one subsystem would have less of an overall effect. Clearly, a significant engineering effort will be required to detail the optimum design for any of the 8 cases under discussion in this section.

DETAILS OF CASE 3 DESIGN

Summary

The Case 3 design bases are:

- -- 3,790 m^3/day (1 x 10^6 gpd) pine or hardwood decker effluents;
- -- tubular membrane assemblies;
- -- hydrasieve and sock filter prefiltration;
- -- three-stage UF system (see Table 17).

Description

The Case 3 system is designed to process either pine decker or hardwood decker effluent at a rate of 3,790 m^3/day (1 MM gpd). Since the decker effluents can be recycled to the weak black liquor with less concentration

TABLE 16. ULTRAFILTRATION SECTION DESIGN -- CASE 2

Item	Stage number 1	 2	 3
Total membrane area, m^2 (ft^2)	1,962 (21,120)	654 (7,040)	654 (7,040)
No. of subsystems	6	2	2
Membrane area per subsystem, m^2 (ft^2)	327 (3,520)	327 (3,520)	327 (3,520)
No. of parallel membrane passes per subsystem	100	100	100
Circulation flowrate per subsystem, m^3/day (gpm)	13,516 (2,480)	13,516 (2,480)	13,516 (2,480)
Pressure drop per parallel pass, atm (psi)	2.38 (35)	2.38 (35)	2.38 (35)
Motor power, kW (hp)	74.6 (100)	74.6 (100)	74.6 (100)
Efficiency, %	80	80	80
Brake power per subsystem, kW (hp)	58.9 (79)	58.9 (79)	58.9 (79)
Design flux, m^3/m^2-day (gfd)	2.87 (70)	2.05 (50)	1.03 (25)
Typical conversion (feed basis), %	67	90	98

TABLE 17. ULTRAFILTRATION SECTION DESIGN -- CASE 3

Item	Stage number		
	1	2	3
Total membrane area, m^2 (ft^2)	981 (10,560)	477.5 (5,140)	117.8 (1,268)
No. of subsystems	3	2	2
Membrane area per subsystem, m^2 (ft^2)	327 (3,520)	238.7 (2,570)	58.9 (634)
No. of parallel membrane passes per subsystem	100	73	18
Circulation flowrate per subsystem, m^3/day, (gpm)	13,516 (2,480)	9,946 (1,825)	2,453 (450)
Pressure drop per parallel pass, atm (psi)	2.38 (35)	2.38 (35)	2.38 (35)
Motor power, kW (hp)	74.6 (100)	55.9 (75)	19.6 (25)
Efficiency, %	80	74	60
Brake power per subsystem, kW (hp)	58.9 (79)	47.0 (63)	14.2 (19)
Design flux, m^3/m^2-day (gfd)	2.87 (70)	2.05 (50)	1.64 (40)
Typical conversion (feed basis), %	67	90	98

than required for caustic extract, an overall system conversion of 95% is acceptable. The lower conversion in the third stage results in a higher projected average flux, 1.64 m^3/m^2-day (40 gfd), and a reduced membrane area requirement, 118 m^2 (1,268 ft^2). Except for the stage 3 differences the Case 3 and Case 1 system designs are alike.

DETAILS OF CASE 4 DESIGN

Summary

- -- 7,580 m^3/day (2 x 10^6 gpd) pine or hardwood decker effluent;
- -- tubular membrane assemblies;
- -- hydrasieve and sock filter prefiltration;
- -- three-stage UF system (see Table 18).

Description

The Case 4 system for processing decker effluents parallels the Case 2 design for caustic extraction filtrate. The difference in these design cases is the reduction in overall system conversion to 95% for the decker effluent processing. The effect of this conversion change is observed in the third stage of the ultrafiltration system where membrane area is reduced to 255 m^2 (2,746 ft^2).

DETAILS OF CASE 5 DESIGN (IDEALIZED SPIRAL-WOUND MODULE SYSTEM)

Summary

The Case 5 design bases are as follows:

- -- 3,790 m^3/day (1 x 10^6 gpd) pine caustic extraction filtrate;
- -- spiral-wound membrane modules with Vexar spacers. Each module is 0.1 m (4 in) in diameter x 0.91 m (36 in) long and contains approximately 2.97 m^2 (32 ft^2) of membrane surface area. Three modules would be connected in series and housed in a single shell.
- -- prefiltration consists of a hydrasieve for fiber removal, a back flushable 5-10μ sock filter and disposable string-wound cartridge filters.
- -- the ultrafiltration system consists of 3 stages. These stages are detailed in Table 19.

Description

The design change from tubular assemblies to spiral-wound modules has minimal effect on overall system operation. Three stages in series are still employed for the ultrafiltration system with interstage and system conversion being similar to those for tubes. Feed recirculation in each stage

TABLE 18. ULTRAFILTRATION SECTION DESIGN -- CASE 4

Item	Stage number 1	2	3
Total membrane area, m^2 (ft^2)	1,962 (21,120)	654 (7,040)	255.1 (2,746)
No. of subsystems	6	2	2
Membrane area per subsystem, m^2 (ft^2)	327 (3,520)	327 (3,520)	127.6 (1,373)
No. of parallel membrane passes per subsystem	100	100	39
Circulation flowrate per subsystem, m^3/day, (gpm)	13,516 (2,480)	13,516 (2,480)	5,314 (975)
Pressure drop per parallel pass, atm (psi)	2.38 (35)	2.38 (35)	2.38 (35)
Motor power, kW (hp)	74.6 (100)	74.6 (100)	37.3 (50)
Efficiency, %	80	80	65
Brake power per subsystem, kW (hp)	58.9 (79)	58.9 (79)	28.3 (38)
Design flux, m^3/m^2-day (gfd)	2.87 (70)	2.05 (50)	1.64 (40)
Typical conversion (feed basis), %	67	90	95

TABLE 19. ULTRAFILTRATION SECTION DESIGN -- CASE 5

Item	Stage number 1	2	3
Total membrane area, m^2 (ft^2)	989.9 (10,656)	481.6 (5,184)	338.9 (3,648)
No. of subsystems	3	2	2
Membrane area per subsystem, m^2 (ft^2)	329.9 (3,552)	240.8 (2,592)	169.4 (1,824)
No. of parallel membrane passes per subsystem	37	27	19
Circulation flowrate per subsystem,m^3/day (gpm)	2,017 (370)	1,472 (270)	1,036 (190)
Pressure drop per parallel pass, atm (psi)	2.04 (30)	2.04 (30)	2.04 (30)
Motor power, kW (hp)	14.9 (20)	11.2 (15)	7.5 (10)
Efficiency, %	60	60	60
Brake power per subsystem, kW (hp)	9.7 (13)	7.5 (10)	4.8 (6.5)
Design flux, m^3/m^2-day (gfd)	2.87 (70)	2.05 (50)	1.03 (25)
Typical conversion (feed basis), %	67	90	98

is 54.5 m^3/day (10 gpm). Permeate and concentrate quality is unchanged as are the reuse applications for these streams. On the pretreatment end of the system an additional filter unit is provided to further protect the spiral-wound modules. This unit consists of string-wound cartridge filters. These 5μ filters are disposable.

Membrane area requirements for the spiral systems (Cases 5 through 8) remain unchanged from those developed for tubular systems. That is, the same flux levels have been assumed in each comparative case. In this manner, "ideal" spiral-wound system case analyses will be developed. "Ideal" spiral-wound module performance may not be achievable given the current state-of-the-art of membrane technology. However, if future spiral systems are to be of interest to the pulp and paper industry long-term reliable operation must be demonstrated. This will require improved module designs, potentially having flux levels equivalent to tubes. Again, the spiral-wound module system designs and economic projections presented in this report reflect "ideal" cases and are not achievable given today's technology.

DETAILS OF CASE 6 DESIGN (IDEALIZED SPIRAL-WOUND MODULE SYSTEM)

Summary

- -- 7,580 m^3/day (2×10^6 gpd) pine caustic extraction filtrate;
- -- spiral-wound modules;
- -- hydrasieve, sock filter and cartridge filter pretreatment;
- -- three-stage UF system (see Table 20).

Description

Case 6 is analogous to Case 2 in that a 7,590 m^3/day (2 MM gpd) caustic extract stream is being processed. The first stage of the spiral-wound system consists of 3 subsystems, each with 660 m^2 (7,104 ft^2) of membrane area. The second and third stages are nearly identical. These stages each have two subsystems with ∿325 m^2 (∿3,500 ft^2) of membrane area. Other than the 10% safety margin in membrane area, no excess capacity has been designed into Stage 2. Therefore, during cleaning cycles (which are potentially of a longer duration than for tubular systems) the overall system conversion may fall below 98%. If the bi-weekly cleaning cycles for each of the Case 6 spiral-wound subsystems should exceed 5 hours, then the ultrafiltration system would be in a cleaning mode more than 10% of the time. Under these circumstances a design change to incorporate more membrane area, and/or more subsystems may be required.

DETAILS OF CASE 7 DESIGN (IDEALIZED SPIRAL-WOUND MODULE SYSTEM)

Summary

- -- 3,790 m^3/day (1×10^6 gpd) pine or hardwood decker effluent;
- -- spiral-wound membrane modules;

TABLE 20. ULTRAFILTRATION SECTION DESIGN -- CASE 6

Item	Stage number		
	1	2	3
Total membrane area, m^2 (ft^2)	1,980 (21,312)	642.1 (6,912)	659.9 (7,104)
No. of subsystems	3	2	2
Membrane area per subsystem, m^2 (ft^2)	659.9 (7,104)	321.1 (3,456)	329.9 (3,552)
No. of parallel membrane passes per subsystem	74	36	37
Circulation flowrate per subsystem, m^3/day (gpm)	4,033 (740)	1,962 (360)	2,017 (370)
Pressure drop per parallel pass, atm (psi)	2.04 (30)	2.04 (30)	2.04 (30)
Motor power, kW (hp)	26.1 (35)	14.9 (20)	14.9 (20)
Efficiency, %	65	60	60
Brake power per subsystem, kW (hp)	17.2 (23)	8.9 (12)	9.7 (13)
Design flux, m^3/m^2-day (gfd)	2.87 (70)	2.05 (50)	1.03 (25)
Typical conversion (feed basis), %	67	90	98

-- hydrasieve, sock filter and cartridge filter pretreatment;

-- three-stage UF system (see Table 21).

Description

The spiral-wound system for treating 3,790 m^3/day (1 MM gpd) of decker effluent is designed to have an overall conversion of 95%. It is identical to the Case 5 design for caustic extraction filtrate except that the third stage contains less membrane area (125 m^2) [1,344 ft^2].

DETAILS OF CASE 8 DESIGN (IDEALIZED SPIRAL-WOUND MODULE SYSTEM)

Summary

-- 7,590 m^3/day (2 x 10^6) pine or hardwood decker effluent;

-- spiral-wound membrane modules;

-- hydrasieve sock filter and cartridge filter pretreatment;

-- three-stage UF system (see Table 22).

Description

The final design case considers treatment of 7,590 m^3/day (2 MM gpd) of decker effluent by spiral-wound modules. Overall conversion is 95%. System pretreatment and operations are the same as described for Case 5.

TABLE 21. ULTRAFILTRATION SECTION DESIGN -- CASE 7

Item	Stage number 1	2	3
Total membrane area, m^2 (ft^2)	989.9 (10,656)	481.6 (5,184)	124.9 (1,344)
No. of subsystems	3	2	2
Membrane area per subsystem, m^2 (ft^2)	329.9 (3,552)	240.8 (2,592)	62.4 (672)
No. of parallel membrane passes per subsystem	37	27	7
Circulation flowrate per subsystem, m^3/day(gpm)	2,017 (370)	1,472 (270)	382 (70)
Pressure drop per parallel pass, atm (psi)	2.04 (30)	2.04 (30)	2.04 (30)
Motor power, kW (hp)	14.9 (20)	11.2 (15)	5.6 (7.5)
Efficiency, %	60	60	40
Brake power per subsystem, kW (hp)	9.7 (13)	7.5 (10)	3.0 (4)
Design flux, m^3/m^2-day (gfd)	2.87 (70)	2.05 (50)	1.64 (40)
Typical conversion (feed basis), %	67	90	95

TABLE 22. ULTRAFILTRATION SECTION DESIGN -- CASE 8

Item	Stage Number 1	2	3
Total membrane area, m^2 (ft^2)	1,980 (21,312)	642.1 (6,912)	267.6 (2,880)
No. of subsystems	3	2	2
Membrane area per subsystem, m^2 (ft^2)	659.9 (7,104)	321.1 (3,456)	133.8 (1,440)
No. of parallel membrane passes per subsystem	74	36	15
Circulation flowrate per subsystem, m^3/day (gpm)	4,033 (740)	1,962 (360)	818 (150)
Pressure drop per parallel pass, atm (psi)	2.04 (30)	2.04 (30)	2.04 (30)
Motor power, kW (hp)	26.1 (35)	14.9 (20)	7.5 (10)
Efficiency, %	65	60	50
Brake power per subsystem, kW (hp)	17.2 (23)	8.9 (12)	3.7 (5)
Design flux, m^3/m^2-day (gfd)	2.87 (70)	2.05 (50)	1.64 (40)
Typical conversion (feed basis), %	67	90	95

SECTION 8

PROJECTED ECONOMICS FOR FULL-SCALE SYSTEMS

INTRODUCTION

Capital and operating cost estimates have been prepared for each of the eight design cases. Because of the significant cost differential between tubular and spiral-wound ultrafiltration systems, the economic analyses which follow will center on tubular systems (design cases 1 through 4) before discussion of spiral-wound systems (design cases 5 through 8) is begun. Included with the tubular system cost estimates are projections of future cost savings due to advances in membrane technology. Note, cost estimates for spiral-wound systems are based on ideal system performance and are not attainable given today's technology.

COSTS FOR DESIGN CASES 1 THROUGH 4

Bases for Capital Cost Projections

The capital costs for the full-scale treatment systems incorporating tubular ultrafiltration systems were derived in the following manner. First, all major auxiliary equipment and the ultrafiltration system were sized. The auxiliary equipment (pretreatment system, permeate collection tank, etc.) was then costed through vendor quotes and catalogs. The ultrafiltration system costs were divided into hardware and membrane costs. Hardware costs for the 3,790 m^3/day (1 MM gpd) caustic extraction filtrate system (case 1) were broken out as carefully as possible for this type of engineering estimate. Hardware costs for the remaining 3 tubular systems were derived as fractions of the case 1 hardware costs. In all case 1 through 4 estimates, membrane costs were held constant at \$293.5/m^2 (\$27.27/ft^2). This corresponds to \$120 per 6.1 m (20 ft) tubular assembly.

The on-site engineering design and installation expenses were calculated as percentages of the equipment costs. Different multipliers were used for the auxiliary equipment engineering and installation than for the ultrafiltration system. This is because the ultrafiltration systems are supplied as skid-mounted subsystems and all engineering internal to the ultrafiltration system (inter-stage piping and electrical, etc.) will have been completed by the vendor. The percentage increases for installation and design used were:

Cost element	Auxiliary equipment	Ultrafiltration system
Installation	40% of equipment cost	15% of equipment cost
Detailed engineering design	12% of installed cost	5% of installed cost

Costs for a building to house each treatment system were calculated by estimating the area requirements (based on subsystem conceptual design) and applying a multiplier of \$161.5/m^2 (\$15/ft^2).

Installed equipment cost, engineering and building costs were totaled and the following three categories and multipliers were used in sequence to obtain the total installed cost.

Cost element	Multiplier
Administration and Supervision	2%
Contingency	10%
Inflation	10%

Design Case 1 Capital Cost

The equipment costs for the 3,790 m^3/day (1 MM gpd) caustic extraction filtrate treatment system are detailed below. The total installed capital cost is calculated in Table 23.

Pretreatment--

Hydrasieve - 3,790 m^3/day (700 gpm) fiberglass frame, 0.76 mm (0.030 in) screen, two 1.83 m (72 in) screens placed back-to-back	\$15,000
Fiber Pump - 16.4 m^3/day (3 gpm), 0.37 kw (0.5 hp)	\$ 700
Feed Pump - 3,790 m^3/day (700 gpm), 4.1 atm (60 psig) head, 29.8 kw (40 hp) motor, carbon steel	\$ 2,500
Feed Pump (Spare) - same as above	\$ 2,500
Sock Filters (2) - 3,790 m^3/day (700 gpm), automatic controls for extended backwash, 0.15 m (6 in) stainless steel drain header, polypropylene sock, 5 to 10μ	\$41,000

TABLE 23. CASE 1 DESIGN CAPITAL COST SUMMARY

Pretreatment system	84,200		
Permeate collection/ distribution subsystem	53,800		
Concentrate collection/ distribution subsystem	10,100	148,100	
Installation @ 40% of total auxiliary equipment cost		59,240	
Auxiliary equipment installed cost			207,340
Ultrafiltration system (includes $524,160 for membranes)		1,349,000	
Ultrafiltration system installation @ 15% of UF system cost		202,350	
Ultrafiltration system installed cost			1,551,350
Total equipment cost			1,758,690
Detailed engineering design @ 12% of auxiliary equipment installed cost and 5% of UF system installed cost			102,448
Building (372 m^2 @ $161/$m^2$) [4000 ft^2 @ $15/$ft^2$]			60,000
Subtotal A			1,921,138
Administration and supervision (2% of subtotal A)			38,423
Subtotal B			1,959,561
Contingency (10% of subtotal B)			195,956
Subtotal C			2,155,517
Inflation (10% of subtotal C)			215,551
Total installed cost			$ 2,371,068

Piping and Valves (rough estimate) - includes 305 m (1,000 ft) of 0.25 m (10 in) diameter carbon steel pipe @ \$32.8/m (\$10/ft)	\$20,000
SUBTOTAL FOR EQUIPMENT	\$81,700
Freight-in transportation costs @ 3% of equipment cost	\$ 2,500
TOTAL PRETREATMENT SYSTEM	\$84,200

(Excluding installation, engineering design, equipment procurement and startup)

Ultrafiltration System--

A total of 1,785 m^2 (19,200 ft^2) of membrane will be used. The costs are broken out by stages and major categories below. Costs shown for the UF system include all factors except on-site engineering, on-site installation, on-site supervision and start-up.

Stage 1 Pumping Packages (3 required) - include 13,516 m^3/day (2,480 gpm) pumps, 74.6 kw (100 hp) motors, all valves, piping and mounting materials, engineering and assembly @ \$38,900	\$116,700
Stage 1 Membrane Racks (3 required) - include inlet and outlet headers, permeate collection headers, membrane supports, all materials, assembly labor @ \$42,900	\$128,700
Stage 1 Automatic Cleaning Packages (3 required) - include piping, solenoid valves, engineering and assembly @ \$9,240 each	\$ 27,720
Stage 2 Pumping Packages (2 required) - include 9,946 m^3/day (1,825 gpm) pumps, 55.9 kw (75 hp) motors, all valves, piping, mounting materials, engineering and assembly @ \$38,900	\$ 77,800
Stage 2 Membrane Racks (2 required) - similar to stage 1 membrane racks @ \$36,300	\$ 72,600
Stage 2 Automated Cleaning Packages (2 required) - similar to stage 1 units @ \$9,240	\$ 18,480

Stage 3 Pumping Package (2 required) - include 6,813 m^3/day (1,250 gpm) pumps, 37.3 kw (50 hp) motors, all valves, piping, mounting materials, heat exchangers in circulation loop, engineering and assembly @ $30,900	$ 61,800
Stage 3 Membrane Racks (2 required) - Similar to stage 1 membrane racks @ $28,200	$ 56,400
Stage 3 Automatic Cleaning Packages (2 required) - similar to stage 1 units @ $7,920	$ 15,840
Cleaning Station - includes 7.58 m^3 (2,000 gal) clean tank, 2.27 m^3 (600 gal) rinse tank, 3,270 m^3/day (600 gpm) pump with 18.6 kw (25 hp) motor, tank and pump mounting, engineering and assembly labor	$ 29,700
Interstage Piping - for feed, permeate and cleaning. Includes pressure control of feed and flow ratio control of feed to concentrate to automatically control system conversion. Materials, engineering and assembly	$128,000
Electrical - includes central control panel with semi-graphic display, subsystem wiring to common terminal box, materials, engineering and assembly	$ 91,100
Ultrafiltration Membranes - tubular, 25.4 mm (1 in) diameter x 3.05 m (10 ft) long WRP membrane assemblies. Including interconnectors, fiberglass backing, plastic shells and U-bends. 8,736 required @ $60 each	$524,160
TOTAL UF SYSTEM COST	$1,349,000

(including all factors except on-site engineering, on-site installation, on-site supervision and start-up)

Permeate Collection/Distribution System--

The UF permeate will be collected in 189.5 m^3 (50,000 gal) tank ($\sim$ 1 hour residence time). From 5% to 50% of the permeate will be returned to the bleachery for reuse. The remaining permeate will be used for sock filter backflush, subsystem cleaning blended with the mill water supply or discharged to sewer.

Permeate Collection Tank - 189.5 m^3 (50,000 gal), construction on-site	$ 30,000
Permeate Distribution Pump - 3,790 m^3/day (700 gpm), 18.6 kw (25 hp) motor, 2.72 atm (40 psig) head, carbon steel	$ 2,200
Piping and Valves (rough estimate) includes 305 m (1,000 ft) of 0.25 m (10 in) diameter carbon steel pipe @ $37.8/m ($10/ft)	$ 20,000
SUBTOTAL FOR EQUIPMENT	$ 52,200
Freight-in transportation costs @ 3% of equipment costs	$ 1,600
TOTAL PERMEATE COLLECTION/DISTRIBUTION SYSTEM	$ 53,800
(Excluding installation, engineering design, equipment procurement and startup)	

Concentrate Collection/Distribution System--

The UF system concentrate will be returned to the weak black liquor, the lime kiln or admixed with the lime sludge at an average flowrate of 76.3 m^3/day (14 gpm).

Concentrate Holding Tank - 1.9 m^3 (500 gal)	$ 1,500
Concentrate Transfer Pump - 76.3 m^3/day (14 gpm), 2.7 atm (40 psig) head, 3.72 kw (5 hp) motor, carbon steel	$ 800
Piping and Valves (rough estimate)	$ 7,500
SUBTOTAL FOR EQUIPMENT	$ 9,800
Freight-in transportation costs @ 3% of equipment costs	$ 300
TOTAL CONCENTRATE COLLECTION/DISTRIBUTION SYSTEM	$ 10,100

Summary--

The total installed cost for the case 1 system is estimated to be $2,371,068 (see Table 23). The ultrafiltration system accounts for 57% of this figure.

The ultrafiltration system cost consists of approximately $\$291/\mathrm{m}^2$ ($\$27.3/\mathrm{ft}^2$) for membranes and $\$463/\mathrm{m}^2$ ($\$43/\mathrm{ft}^2$) for hardware. Overall, this amounts to $\$753/\mathrm{m}^2$ ($\$70/\mathrm{ft}^2$) for the 178.6 m^2 (19,220 ft^2) ultrafiltration system.

Design Case 2 Capital Cost

Pretreatment--

The capacity of the Case 1 pretreatment system must be doubled to treat 7580 m^3/day (2 MM gpd) of caustic extraction filtrate. The costs for the increased capacity components are shown below. The general characteristics of the components are the same as detailed in the Case 1 cost discussion.

Hydrasieve - 7,630 m^3/day (1,400 gpm)	$ 25,000
Fiber pump - 7,630 m^3/day (6 gpm)	$ 1,200
Feed pump - 7,630 m^3/day (1,400 gpm)	$ 4,000
Feed pump (spare) - 7,630 m^3/day (1,400 gpm)	$ 4,000
Sock filters (2) - 7,630 m^3/day (1,400 gpm)	$ 82,000
Piping and valves -	$ 35,000
SUBTOTAL FOR EQUIPMENT	$151,200
Freight-in transportation costs @ 3% of equipment cost	$ 4,500
TOTAL PRETREATMENT SYSTEM	$155,700
(Excluding installation, engineering design, equipment procurement and startup)	

The Case 2 pretreatment system cost is 1.85 x the Case 1 cost estimate.

Ultrafiltration System--

The hardware cost for the ultrafiltration system is based on a system consisting of 10 identical subsystems. Each subsystem would be the same as described for stage 1 of the case 1 system.

UF system hardware -	$1,317,600

UF membranes - 3,270 m^2 (35,200 ft^2) @ \$293.5/m^2 (\$27.27/ft^2)	960,000
TOTAL UF SYSTEM COST	\$2,277,600

(including all factors except on-site installation, on-site supervision and startup)

This ultrafiltration system provides a doubling of process capacity for 1.69X the case 1 cost.

Permeate Collection/Distribution System--

For the 7,580 m^3/day (2 MM gpd) systems (cases 2 and 4) it was decided to retain the same size permeate holding tank as for the 3,790 m^3/day (1 MM gpd) systems. This volume (189.5 m^3) [50,000 gal] would still be sufficient for flushing, cleaning and recycling operations. Holding tank residence time would be reduced to 35 minutes.

Permeate collection tank- 189.5 m^3 (50,000 gal)	\$ 30,000
Permeate distribution pump - 7,630 m^3/day (1,400 gpm)	\$ 4,000
Piping and valves	\$ 35,000
SUBTOTAL FOR EQUIPMENT	\$ 69,000
Freight-in transportation costs @ 3% of equipment cost	\$ 2,100
TOTAL PERMEATE COLLECTION/DISTRIBUTION SYSTEM	\$ 71,000

(excluding installation, engineering design, equipment procurement and startup)

Concentrate Collection/Distribution System--

Concentrate holding tank - 1.9 m^3 (500 gal) (same size as for Case 1)	\$ 1,500
Concentrate transfer pump - 153 m^3/day (28 gpm)	\$ 1,000
Piping and Valves	\$ 11,500
SUBTOTAL FOR EQUIPMENT	\$ 14,000

Freight-in transportation costs @ 3% of equipment costs	$ 400
TOTAL CONCENTRATE COLLECTION/DISTRIBUTION SYSTEM	$ 14,400

(excluding installation, engineering design, equipment procurement and startup)

Summary--

The case 2 capital cost is summarized in Table 24. Total installed cost is $3,972,151. This cost is 1.675X the case 1 installed cost, the ultrafiltration system accounts for 57%. The ultrafiltration system is \$696/m^2 (\$64.70/ft^2) (\$402.6/m^2 for hardware and \$293.5/m^2 for membranes) [\$37.4/ft^2 for hardware and \$27.3/ft^2 for membranes].

Capital Cost for Design Case 3

The case 3 system is designed to treat 3,790 m^3/day (1 MM gpd) pine or hardwood decker effluent. The only major change from the case 1 design is that processing is only necessary to 95% conversion in the ultrafiltration system. Therefore the third stage of the ultrafiltration system contains less membrane area than the corresponding stage under case 1.

Pretreatment System--

The pretreatment system is identical to that presented for case 1.

TOTAL PRETREATMENT SYSTEM COST	$ 84,200

Ultrafiltration System--

The ultrafiltration system hardware cost is unchanged from case 1 for stage 1, stage 2 and interstage connections. The stage 3 hardware cost will be somewhat reduced. Overall hardware cost is calculated as the same hardware cost factor (\$463/m^2) [\$43/ft^2] as for case 1.

UF system hardware -	$729,624
UF membranes - 1,576 m^2 (16,968 ft^2) @ \$295.5/m^2 (\$27.27/ft^2)	$462,760
TOTAL UF SYSTEM COST	$1,192,384

TABLE 24. CASE 2 DESIGN CAPITAL COST SUMMARY

Pretreatment system	155,700		
Permeate collection/ distribution subsystem	71,100		
Concentrate collection/ distribution subsystem	14,400		
		241,200	
Installation @ 40% of total auxiliary equipment cost		96,480	
Auxiliary equipment installed cost			337,680
Ultrafiltration system (includes $960,000 for membranes)		2,277,600	
Ultrafiltration system installation @ 15% of UF system cost		341,640	
Ultrafiltration system installed cost			2,619,240
Total equipment cost			2,956,920
Detailed engineering design @ 12% of auxiliary equipment installed cost and 5% of UF system installed cost			171,480
Building ($557 m^2 @ $161/$m^2$) [6,000 ft^2 @ $15/$ft^2$]			90,000
Subtotal A			3,218,400
Administration and supervision (2% of subtotal A)			64,370
Subtotal B			3,282,770
Contingency (10% of subtotal B)			328,277
Subtotal C			3,611,047
Inflation (10% of subtotal C)			361,104
Total installed cost			$ 3,972,151

Permeate and Concentrate Collection/Distribution Systems--

No change from the case 1 study occurs for either the permeate or concentrate collection/distribution system.

TOTAL PERMEATE COLLECTION/DISTRIBUTION SYSTEM	\$ 53,800
TOTAL CONCENTRATE COLLECTION/DISTRIBUTION SYSTEM	\$ 10,100

Summary--

Table 25 details the case 3 capital cost projection. Total installed cost is estimated to be \$2,137,736. Membrane system cost is 56% of total cost. As with case 1, ultrafiltration system cost is \$753.5/m^2 (\$70/ft^2).

Capital Cost for Design Case 4

Design case 4 is for 7,580 m^3/day (2 MM gpd) decker effluent treatment. The costs parallel case 2 costs with a reduction in ultrafiltration system costs consistent with the reduced membrane area.

Pretreatment System--

The pretreatment system is identical to the case 2 pretreatment system.

TOTAL PRETREATMENT SYSTEM COST	\$155,700

Ultrafiltration System--

UF system hardware - 2,871 m^2 (30,906 ft^2) @ \$402.6/m^2 (\$37.4/ft^2)	\$1,155,900
UF membranes - 2,871 m^2 (30,906 ft^2) @ \$293.5/m^2 (\$27.27/ft^2)	\$ 842,810
TOTAL UF SYSTEM COST	\$1,998,710

Permeate and Concentrate Collection/Distribution Systems--

Permeate and concentrate collection/distribution system costs are unchanged from those presented for case 2.

TOTAL PERMEATE COLLECTION/DISTRIBUTION SYSTEM	\$ 71,100
TOTAL CONCENTRATE COLLECTION/DISTRIBUTION SYSTEM	\$ 14,400

TABLE 25. CASE 3 DESIGN CAPITAL COST SUMMARY

Pretreatment system	84,200		
Permeate collection/ distribution subsystem	53,800		
Concentrate collection/ distribution subsystem	10,100	148,100	
Installation @ 40% of total auxiliary equipment cost		59,240	
Auxiliary equipment installed cost			207,340
Ultrafiltration system (includes $462,760 for membranes)		1,192,400	
Ultrafiltration system installation @ 15% of UF system cost		178,860	
Ultrafiltration system installed cost			1,371,260
Total equipment cost			1,578,600
Detailed engineering design @ 12% of auxiliary equipment installed cost and 5% of UF system installed cost			93,444
Building (372 m^2 @ \$161/m^2) [4000 ft^2 @ \$15/ft^2]			60,000
Subtotal A			1,732,044
Administration and supervision (2% of subtotal A)			34,681
Subtotal B			1,766,725
Contingency (10% of Subtotal B)			176,672
Subtotal C			1,943,397
Inflation (10% of subtotal C)			194,339
Total installed cost			$ 2,137,736

Summary--

The case 4 design capital cost summary is shown in Table 26. Total installed cost is \$3,556,524. Again, membrane system cost is 56% of total cost. As with case 2, the ultrafiltration system is \$696.4/m^2 (\$64.70/ft^2) of membrane area.

Bases for Operating Cost Projections

Operating costs fall into 3 main categories: materials, conversion expense less depreciation, and depreciation. The only materials required for system operation are cleaning chemicals for the membrane system. These chemicals are caustic and EDTA. They were costed at bulk quantity prices.

Conversion expense (less depreciation) is made up of four major elements. The first element, labor, was assumed at 6 hours per day for all systems. Current labor expense (including fringe benefits) is \$10/hour. Electrical power was calculated based on feed pump, ultrafiltration system circulation pumps and permeate distribution pump brake horsepower. The current Canton Mill power cost of \$0.0225/kwh was used. Repair and maintenance costs consist of materials and labor. Maintenance material was calculated as 1.5% of the hard goods cost (total equipment cost less membrane replacement cost). The maintenance labor cost was assumed to be equivalent to the material cost. Thus, maintenance labor hours were back-calculated using a rate of \$15/hour. The final element for this cost category is insurance and taxes. This operating cost was assumed to be one-half of the maintenance material cost.

Depreciation expense has two elements: membrane replacement cost and equipment amortization. The tubular membranes were depreciated over a 3 year life at their replacement cost, not their original cost. This replacement cost is \$172.2/m^2 (\$16/ft^2) when ordered in minimum quantities of 2,000 3.05 m (10 ft) lengths. This reduction in tubular assembly original cost (\$27.3/ft^2) results from return (and recovery) of the permeate collection shell and the fiberglass support tube. Also, replacement U-bends are not required. The equipment (other facilities) was depreciated over 15 years on a straight line basis.

Throughout the operating cost estimates the plant was assumed to be operating 24 hours per day, 365 days per year.

Operating Costs for Design Case 1

The case 1 operating costs are detailed below with sample calculations.

Materials--

The cleaning materials are 0.5% caustic and 0.25% EDTA. A cleaning solution volume of 7.58 m^3 (2,000 gal) per subsystem is required. This

TABLE 26. CASE 4 DESIGN CAPITAL COST SUMMARY

Pretreatment system	155,700		
Permeate collection/ distribution subsystem	71,100		
Concentrate collection/ distribution subsystem	14,400	241,200	
Installation @ 40% of total auxiliary equipment cost		96,480	
Auxiliary equipment installed cost			337,680
Ultrafiltration system (includes $842,810 for membranes)		1,998,710	
Ultrafiltration system installation @ 15% of UF system cost		299,807	
Ultrafiltration system installed cost			2,298,517
Total equipment cost			2,636,197
Detailed engineering design @ 12% of auxiliary equipment installed cost and 5% of UF system installed cost			155,447
Building (557 m^2 @ $161/$m^2$) [6,000 ft^2 @ $15/$ft^2$]			90,000
Subtotal A			2,881,644
Administration and supervision (2% of subtotal A)			57,633
Subtotal B			2,939,277
Contingency (10% of subtotal B)			293,927
Subtotal C			3,233,204
Inflation (10% of subtotal C)			323,320
Total installed cost			$ 3,556,524

volume is held constant even though subsystem size varies because the difficulty of cleaning may increase in the higher conversion stages. One cleaning per subsystem biweekly is projected.

For caustic,

$$\frac{2{,}000\text{ gal}}{\text{cleaning}}\;\bigg|\;\frac{3.77\text{ kg}}{\text{gal}}\;\bigg|\;\frac{1\text{ cleaning}}{2\text{ days}}\;\bigg|\;\frac{0.5\%\text{ caustic}}{}\;=\;18.8\text{ kg caustic/day}$$

If a 50% caustic soda is used 37.7 kg/day (83 lb/day) are required. At a cost of \$0.15/kg (\$0.07/lb) of caustic soda a daily cost of \$5.81 is incurred.

For EDTA,

$$\frac{2{,}000\text{ gal}}{\text{cleaning}}\;\bigg|\;\frac{3.77\text{ kg}}{\text{gal}}\;\bigg|\;\frac{1\text{ cleaning}}{2\text{ days}}\;\bigg|\;\frac{0.25\%\text{ caustic}}{}\;=\;9.44\text{ kg EDTA/day}$$

At a cost of \$1.95/kg (\$0.885/lb) the daily charge is \$18.41.

Total material cost is \$24.22/day.

Conversion Expense Less Depreciation--

Operating Labor--

$$\frac{6\text{ hr}}{\text{day}}\;\bigg|\;\frac{\$10}{\text{hr}}\;=\;\$60\text{/day}$$

Electrical Power--

$$\frac{500\text{ hp}}{}\;\bigg|\;\frac{.7457\text{ kw}}{\text{hp}}\;\bigg|\;\frac{24\text{ hr}}{\text{day}}\;\bigg|\;\frac{\$0.0225}{\text{kwh}}\;=\;\$203.74\text{/day}$$

Maintenance Material--

$$\frac{1.5\%}{}\;\bigg|\;\frac{(\$1{,}758{,}690-307{,}200)}{\text{year}}\;\bigg|\;\frac{\text{year}}{365\text{ days}}\;=\;\$59.64\text{/day}$$

Maintenance Labor--

$$\frac{4\text{ hr}}{\text{day}}\;\bigg|\;\frac{\$15}{\text{hr}}\;=\;\$60\text{/day}$$

Insurance and Taxes--

$$\frac{0.5}{}\;\bigg|\;\frac{\$59.64}{\text{day}}\;=\;\$29.82\text{/day}$$

Total conversion expense less depreciation is \$413.20/day.

Depreciation--

Membranes--

$$\frac{1{,}784\ \text{m}^2}{3\ \text{years}}\ \Bigg|\ \frac{\$172.2}{\text{m}^2}\ \Bigg|\ \frac{\text{year}}{365\ \text{days}} = \$280.55/\text{day}$$

Other Facilities--

$$\frac{\$2{,}063{,}868}{15\ \text{years}}\ \Bigg|\ \frac{\text{year}}{365\ \text{days}} = \$376.96/\text{day}$$

Total depreciation is $657.51/day.

Summary--

The case 1 operating costs are summarized in Table 27 on daily, yearly, volume and percentage bases. Overall operating cost is $1,094.93/day ($0.29/m^3) [$1.10/1000 gal]. For a 727 metric ton/day (800 ton/day) pulp mill this amounts to a cost of $1.51/metric ton ($1.37/ton) of pine pulp.

The major cost factors are power - 18.2%, membrane replacement - 25.5%, and facility depreciation - 34.5%. Total depreciation accounts for 60% of the operating cost.

Operating Costs for Design Case 2

The same volume (7.58 m^3) [2,000 gal] cleaning solution is used with 10 subsystems being cleaned per 2-week period rather than 7 as in case 1. Material costs thus increase by 10/7.

All conversion expenses are figured in a like manner to the sample calculations presented above for case 1. These expenses are summarized in Table 28.

For this 7,580 m^3/day (2 MM gpd) caustic extraction filtrate treatment system daily operating costs are $1,835.03. This is equivalent to $0.24/m^3 ($0.92/1000 gal) treated or $2.52/metric ton (727 metric ton basis) [$2.29/ton (800 ton basis)]. Major cost factors are again power, membrane replacement and facilities depreciation. These factors account for 19.6%, 28.3% and 33.7% of the operating costs, respectively.

Operating Costs for Design Case 3

Case 3 operating costs are detailed in Table 29. Daily costs are $998.01 for washing of 1,318 metric tons/day (1,450 tons/day) of mixed pulp production. This translates to $0.26/m^3 ($1.00/1000 gal) or $0.76/metric ton ($0.69/ton) of pulp.

TABLE 27. CASE 1 DESIGN OPERATING COST DATA

Cost Element	Quantity	Unit Cost	$/day	$/year	$/m^3	$/M-gal	% of Total
Material (for cleaning)							
Caustic (50%)	37.6 kg/day	$ 0.154/kg	5.81	2,121			
EDTA	4.9 kg/day	1.95/kg	18.41	6.720			
Total materials			24.22	8,841	0.006	0.03	2.7
Conversion expense							
Labor (including benefits)	6 hr/day	$ 10/hr	60.00	21,900	0.016	0.06	5.5
Electrical power	9,055 kwh/day	$0.0225/kwh	203.74	74,365	0.054	0.20	18.2
Repair and Maintenance							
Material	1.5% of total equipment less membrane replacement cost yearly - (1.5% x 1,451,170)/yr		59.64	21,768	0.158	0.06	5.5
Labor	4 hr/day	$ 15/hr	60.00	21,900	0.016	0.06	5.5
Insurance and Taxes	0.5 x maintenance materials		29.82	10,884	0.079	0.03	2.7
Total, excluding depreciation			413.20	150,817	0.109	0.41	37.3
Depreciation							
Membranes	$ 307,200	3-yr life	280.55	102,400	0.074	0.28	25.5
Other facilities	$ 2,063,868	15-yr life	376.96	137,591	0.997	0.38	34.5
Total conversion expense			$ 1,070.71	$ 390,808	0.283	1.07	97.3
Total incremental cost			$ 1,094.93	$ 399,649	0.289	1.10	100.0

TABLE 28. CASE 2 DESIGN OPERATING COST DATA

Cost Element	Quantity	Unit Cost	$/day	$/year	$/m³	$/M-gal	% of Total
Material (for cleaning)							
Caustic (50%)	53.8 kg/day	$ 0.154/kg	8.31	3,033			
EDTA	13.5 kg/day	1.95/kg	26.28	9,592			
Total materials			34.59	12,625	0.005	0.02	2.2
Conversion expense							
Labor (including benefits)	6 hr/day	$ 10/hr	60.00	21,900	0.008	0.03	3.3
Electrical power	15,929 kwh/day	$ 0.0025/kwh	358.40	130,816	0.047	0.18	19.6
Repair and Maintenance							
Material	1.5% of total equipmment less membrane replacement cost yearly = (1.5% x 2,393,720)/yr		98.37	35,905	0.013	0.05	5.4
Labor	6.5 hr/day	$ 15/hr	97.50	35,588	0.013	0.05	5.4
Insurance and Taxes	0.5 x maintenance material		49.19	17,954	0.006	0.02	2.2
Total, excluding depreciation			663.46	242,163	0.088	0.33	35.9
Depreciation							
Membranes	$ 563,200	3-yr life	514.34	187,734	0.068	0.26	28.3
Other facilities	$ 3,408,951	15-yr life	622.64	227,264	0.082	0.31	33.7
Total conversion expense			$ 1,800.44	657,161	0.238	0.90	97.8
Total incremental cost			$ 1,835.03	669,786	0.242	0.92	100.0

TABLE 29. CASE 3 DESIGN OPERATING COST DATA

Cost Element	Quantity	Unit Cost	$/day	$/year	$/m^3	$/M-gal	% of Total
Material (for cleaning)							
Caustic (50%)	37.6kg/day	$ 0.154/kg	5.81	2,121			
EDTA	4.9 kg/day	1.95/kg	18.41	6,720			
Total materials			24.22	8,841	0.006	0.03	3.0
Conversion expense							
Labor (including benefits)	6 hr/day	$ 10/hr	60.00	21,900	0.016	0.06	6.0
Electrical power	8,124 kwh/day	$ 0.0025/kwh	182.79	66,718	0.048	0.18	18.0
Repair and Maintenance							
Material	1.5% of total equipmment less membrane replacement cost yearly = (1.5% x 1,307,112)/yr		53.72	19,607	0.014	0.05	5.0
Labor	4 hr/day	$ 15/hr	60.00	21,900	0.016	0.06	6.0
Insurance and Taxes	0.5 x maintenance material		28.48	10,395	0.075	0.03	3.0
Total, excluding depreciation			384.99	140,520	0.102	0.38	38.0
Depreciation							
Membranes	$ 271,488	3-yr life	247.93	90,496	0.066	0.25	25.0
Other facilities	$ 1,866,248	15-yr life	340.87	124,417	0.090	0.34	34.0
Total conversion expense			$ 973.79	355,435	0.258	0.97	97.0
Total incremental cost			$ 998.01	364,274	0.264	1.00	100.0

Operating Costs for Design Case 4

Table 30 presents case 4 operating costs. Total daily costs are \$1,663.24 or \$0.22/m^3 (\$0.83/1000 gal). Operating cost is \$1.26/metric ton (\$1.15/ton) of pulp (1,378 metric ton/day basis) [1,450/ton day basis].

Summary of Capital and Operating Costs for Design Cases 1 through 4

A summary of the projected economics for treatment systems incorporating tubular ultrafiltration systems is given in Table 31. Capital investment for all cases ranges from \$2 to \$4 MM. Treatment costs, based on 727 metric tons of pine pulp produced per day, range from \$1.51 to \$2.52 for caustic extraction filtrate. The treatment costs range from \$0.76 to \$1.26 for decker effluents (1,318 metric ton/day basis).

In the case 1 through 4 economics the membrane replacement cost is a minimum of 25% of the operating cost. This is with a projected membrane life of 3 years. If membrane life were, in fact, only 2 years the operating costs would increase by \$0.19/metric ton for case 1, \$0.35/metric ton for case 2, \$0.09/metric ton for case 3 and \$0.17/metric ton for case 4.

Future Costs for Tubular Ultrafiltration Systems

Future technological advances are expected to reduce large-scale tubular ultrafiltration system capital costs. These advances will take the form of lower-cost, more-compact modules, possibly with insertable membranes. The insertable membranes would have a lower replacement cost than current membranes. Lower cost systems may be available in the next 2 to 5 years.

To assess the impact of reduced future ultrafiltration system costs and lower membrane replacement costs on overall treatment system capital and operating costs the case 1, 3,790 m^3/day (1 MM gpd) caustic extraction filtrate system, has been reanalyzed with the following changes:

Capital costs

-- auxiliary equipment and building costs increased 20%;

-- UF system cost calculated at \$646/m^2 (\$60/ft^2).

Operating costs

-- cleaning chemicals expense increased 10%;

-- labor expense increased 20%;

-- electrical power costed at \$0.025/kwh;

-- membrane replacement costed at \$86.1/m^2 (\$8/ft2).

The revised capital and operating cost summaries are presented in Tables 32 and 33, respectively.

TABLE 30. CASE 4 DESIGN OPERATING COST DATA

Cost Element	Quantity	Unit Cost	$/day	$/year	$/m^3	$/M-gal	% of Total
Material (for cleaning)							
Caustic (50%)	53.8 kg/day	$ 0.154/kg	8.31	3,033			
EDTA	13.5 kg/day	1.95/kg	26.28	9,592			
Total materials			34.59	12,625	0.005	0.02	2.4
Conversion expense							
Labor (including benefits)	6 hr/day	$ 10/hr	60.00	21,900	0.008	0.03	3.6
Electrical power	14,460 kwh/day	$ 0.0025/kwh	325.40	118,771	0.043	0.16	19.3
Repair and Maintenance							
Material	1.5% of total equipmment less membrane replacement cost yearly = (1.5% x 2,141,701)/yr		88.02	32,126	0.012	0.04	4.8
Labor	6.5 hr/day	$ 15/hr	97.50	35,568	0.013	0.05	6.0
Insurance and Taxes	0.5 x maintenance material		46.87	17,108	0.006	0.02	2.4
Total, excluding depreciation			617.79	225,493	0.082	0.30	36.1
Depreciation							
Membranes	$ 494,496	3-yr life	451.59	164,832	0.059	0.23	27.7
Other facilities	$ 3,062,028	15-yr life	559.27	204,135	0.074	0.28	33.7
Total conversion expense			$ 1,628.65	594,457	0.215	0.81	97.6
Total incremental cost			$ 1,663.24	607,082	0.219	0.83	100.0

TABLE 31. SUMMARY OF PROJECTED ECONOMICS FOR DESIGN CASES 1 THROUGH 4

Item	Design case number			
	1	2	3	4
Stream	Caustic extraction filtrate	Caustic extraction filtrate	Decker effluent	Decker effluent
Flow, m^3/day (MM gal/day)	3,790(1)	7,580(2)	3,790(1)	7,580(2)
UF membrane cost, $	524,160	960,000	462,760	842,810
UF system cost (including membranes), $	1,349,000	2,277,600	1,192,400	1,998,710
Total installed cost, $	2,371,068	3,972,151	2,137,736	3,556,524
Daily operating cost, $	1,095	1,835	998	1,663
Cost per m^3, $	0.29	0.24	0.26	0.22
Cost per 1000 gal, $	1.10	0.92	1.00	0.83
Cost per metric ton of pulp, $	1.51	2.52	0.78	1.31
Cost per ton of pulp, $	1.37	2.29	0.71	1.19

TABLE 32. CASE 1 DESIGN CAPITAL COST SUMMARY WITH FUTURE REDUCTIONS IN ULTRAFILTRATION SYSTEM COSTS CONSIDERED

Pretreatment system	101,040		
Permeate collection/ distribution subsystem	64,560		
Concentrate collection/ distribution subsystem	12,120		
		177,720	
Installation @ 40% of total auxiliary equipment cost		71,088	
Auxiliary equipment installed cost			248,808
Ultrafiltration system (includes \$524,160 for membranes)		1,144,000	
Ultrafiltration system installation @ 15% of UF system cost		171,600	
Ultrafiltration system installed cost			1,315,600
Total equipment cost			1,564,408
Detailed engineering design @ 12% of auxiliary equipment installed cost and 5% of UF system installed cost			87,057
Building (372 m^2 @ \$194/m^2) [4000 ft^2 @ \$18/ft^2]			72,000
Subtotal A			1,723,465
Administration and supervision (2% of subtotal A)			34,469
Subtotal B			1,757,934
Contingency (10% of subtotal B)			175,793
Subtotal C			1,933,727
Inflation (10% of subtotal C)			193,372
Total installed cost			\$ 2,127,099

TABLE 33. CASE 1 DESIGN OPERATING COST DATA WITH FUTURE REDUCTIONS IN ULTRAFILTRATION SYSTEM COSTS CONSIDERED

Cost Element	Quantity	Unit Cost	\$/day	\$/year	\$/m^3	\$/M-gal	% of Total
Material (for cleaning)							
Caustic (50%)	37.6 kg/day	\$ 0.154/kg	6.39	2,333			
EDTA	4.9 kg/day	1.95/kg	20.26	7,395			
Total materials			26.65	9,728	0.007	0.03	3.1
Conversion expense							
Labor (including benefits)	6 hr/day	\$ 12/hr	72.00	26,280	0.019	0.07	7.1
Electrical power	9,055 kwh/day	0.025/kwh	226.38	86,627	0.060	0.23	23.5
Repair and Maintenance							
Material	1.5% of total equipment less membrane replacement cost yearly = (1.5% x 1,410,808)/yr		57.98	21,162	0.015	0.06	6.1
Labor	4 hr/day	\$ 18/hr	72.00	26,280	0.019	0.07	7.1
Insurance and Taxes	0.5 x maintenance material		28.99	10,581	0.008	0.03	3.1
Total, excluding depreciation			457.35	166,933	0.121	0.46	46.9
Depreciation							
Membranes	\$ 153,600	3-yr life	140.27	51,200	0.037	0.14	14.3
Other facilities	\$ 1,973,499	15-yr life	360.46	131.567	0.095	0.36	36.7
Total conversion expense			958.08	349,699	0.253	0.96	97.9
Total incremental cost			\$ 984.73	359,427	0.261	0.98	100.0

The future case 1 system total installed cost becomes $2,127,099 a 10% reduction from the estimated cost based on current technology. The future operating cost is $984.73/day. This is equivalent to $\$0.25/m^3$ ($0.93/1000 gal) or $1.35/metric ton ($1.23/ton) of pulp. This also represents a 10% cost savings over current projections.

The uncertainties associated with the future costs of labor, materials and equipment make it difficult to extrapolate too many years ahead. It may be that reduced ultrafiltration system costs and lower membrane replacement costs will, rather than produce overall cost savings, just counteract other increases to hold down costs to the projections shown in Tables 23 to 30.

COSTS FOR DESIGN CASES 5 THROUGH 8 (IDEALIZED SPIRAL-WOUND MODULE SYSTEMS)

Bases for Capital Cost Projections

Capital cost estimates for full-scale treatment system employing spiral-wound modules were calculated using procedures similar to those described for the tubular-based systems. Differences from the procedures described for the tubular-based systems are listed below.

- -- System costs averaged $\$236.8/m^2$ ($\$22/ft^2$) of membrane area.
- -- Installation costs for the spiral-wound systems were calculated as 40% of the equipment cost. These systems will be of similar complexity to the tubular systems and will require similar installation. Because they have a lower capital cost than tubular systems they require larger multipliers to present realistic associated costs.
- -- Detailed engineering design costs include 12% of the UF system cost. The reasoning for this change is the same as described above for installation costs.

Design 5 Capital Cost (Idealized Spiral-Wound Module System)

Pretreatment--

Pretreatment requirements for design case 5 (and all spiral-wound design cases) are identical to those described for tubular systems since the additional prefiltration required (cartridge filters) is included in the ultrafiltration system capital cost. The case 5 pretreatment is therefore analogous to the case 1 pretreatment.

TOTAL PRETREATMENT SYSTEM COST	$ 84,200

Ultrafiltration System--

The compact nature of spiral-wound systems, as compared to tubular systems, greatly reduces hardware costs. Overall hardware cost for the case 5 ultrafiltration system is $\$164.7/m^2$ ($\$15.30/ft^2$).

UF system hardware -	$298,200
UF membranes - 1,810 m^2 (19,488 ft^2) @ $67.3/m^2 ($6.25/ft^2)	$121,800
TOTAL UF SYSTEM COST	$420,000

Permeate and Concentrate Collection/Distribution Systems--

No change from the case study occurs for either the permeate or concentrate collection/distribution system.

TOTAL PERMEATE COLLECTION/DISTRIBUTION SYSTEM	$ 53,800
TOTAL CONCENTRATE COLLECTION/DISTRIBUTION SYSTEM	$ 10,100

Summary--

Table 34 details the case 5 capital costs. Total installed cost is $1,136,426. The ultrafiltration system cost makes up 37% of this total.

Design Case 6 Capital Cost (Idealized Spiral-Wound Module System)

Pretreatment System--

The pretreatment system is the same as for the 7,580 m^3/day (2 MM gpd) case 2 system.

TOTAL PRETREATMENT SYSTEM COST	$155,700

Ultrafiltration System--

UF System Hardware - 3,282 m^2 (35,328 ft^2) @ $176.5/m^2 ($16.4/ft^2)	$579,200
UF Membranes - 3,282 m^2 (35,328 ft^2) @ $67.3/m^2 ($6.25/ft^2)	$220,800
TOTAL UF SYSTEM COST	$800,000

Permeate and Concentrate Collection/Distribution Systems--

These system components are the same as for the case 2 design.

TOTAL PERMEATE COLLECTION/DISTRIBUTION SYSTEM	$ 71,100
TOTAL CONCENTRATE COLLECTION/DISTRIBUTION SYSTEM	$ 14,400

TABLE 34. CASE 5 DESIGN CAPITAL COST SUMMARY*

Pretreatment system	84,200		
Permeate collection/ distribution subsystem	53,800		
Concentrate collection/ distribution subsystem	10,100		
		148,100	
Installation @ 40% of total auxiliary equipment cost		59,240	
Auxiliary equipment installed cost			207,340
Ultrafiltration system (includes \$121,800 for membranes)		420,000	
Ultrafiltration system installation @ 40% of UF system cost		168,000	
Ultrafiltration system installed cost			588,000
Total equipment cost			795,340
Detailed engineering design @ 12% of auxiliary equipment installed cost and 12% of UF system installed cost			95,440
Building (186 m^2 @ \$161/m^2) [2000 ft^2 @ \$15/ft^2]			30,000
Subtotal A			920,780
Administration and supervision (2% of subtotal A)			18,416
Subtotal B			939,196
Contingency (10% of subtotal B)			93,919
Subtotal C			1,033,115
Inflation (10% of subtotal C)			103,311
Total installed cost			\$ 1,136,426

* Based on idealized spiral-wound modules. Not attainable with current technology.

Summary--

The case 6 capital costs are summarized in Table 35. Total installed cost is $2,061,239. The ultrafiltration system is 39% of this capital cost.

Design Cases 7 and 8 Capital Costs (Idealized Spiral-Wound Module Systems)

Capital costs for design cases 7 and 8, 3,790 m^3/day (1 MM gpd) and 7,580 m^3/day (2 MM gpd) spiral-wound systems for decker effluents, follow directly from the 6 cases previously discussed. These costs are developed in Tables 36 and 37. Total installed costs for case 7 are $1,039,666; for case 8, $1,867,716.

Bases for Operating Cost Projections

The operating costs for the spiral-wound systems differ from those of the tubular systems in that:

- cartridge filter replacement costs are included in the materials. A flowrate of 81.75 m^3/day (15 gpm) per 0.51 m (20 in) cartridge is assumed. Each cartridge is projected to have a 72-hour life. Cartridges cost $4 each.
- module life (membrane replacement life) is 1 year.
- membrane replacement cost is $67.3/$m^2$ ($6.25/$ft^2$) ($200/module, minimum order quantity is 250 modules).

Operating Costs for Design Cases 5 through 8

The operating costs for the treatment systems incorporating spiral-wound modules are detailed in Tables 38 through 41. As with the tubular systems, depreciation accounts for the major portion of the operating costs. The 1-year module life results in membrane replacement costs being >40% of operating costs. Other facility depreciation contributes nearly 25% to total operating costs.

Summary of Capital and Operating Costs for Design Cases 5 through 8

A summary of projected economics for treatment systems based on spiral-wound module technology is presented in Table 42. In reviewing this table and all cost data presented in this section it must be remembered that these costs are based on ideal spiral-wound ultrafiltration systems. While useful for comparison purposes among themselves and with tubular-based systems, the economics given in Table 42 have no intrinsic value. Spiral-wound systems currently cannot process the pulp mill effluent streams at the flux levels on which these economics are based.

TABLE 35. CASE 6 DESIGN CAPITAL COST SUMMARY*

Pretreatment system	155,700		
Permeate collection/ distribution subsystem	71,100		
Concentrate collection/ distribution subsystem	41,400		
		241,200	
Installation @ 40% of total auxiliary equipment cost		96,480	
Auxiliary equipment installed cost			337,680
Ultrafiltration system (includes $220,800 for membranes)		800,000	
Ultrafiltration system installation @ 40% of UF system cost		320,000	
Ultrafiltration system installed cost			1,120,000
Total equipment cost			1,457,680
Detailed engineering design @ 12% of auxiliary equipment installed cost and 12% of UF system installed cost			174,922
Building (232 m^2 @ \$161/m^2) [2,500 ft^2 @ \$15/ft^2]			37,500
Subtotal A			1,670,102
Administration and supervision (2% of subtotal A)			33,402
Subtotal B			1,703,504
Contingency (10% of subtotal B)			170,350
Subtotal C			1,873,854
Inflation (10% of subtotal C)			187,385
Total installed cost			$ 2,061,239

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* Based on idealized spiral-wound modules. Not attainable with current technology.

TABLE 36. CASE 7 DESIGN CAPITAL COST SUMMARY*

Pretreatment system	84,200		
Permeate collection/ distribution subsystem	53,800		
Concentrate collection distribution subsystem	10,100		
		148,100	
Installation @ 40% of total auxiliary equipment cost		59,240	
Auxiliary equipment installed cost			207,340
Ultrafiltration system (includes $107,400 for membranes)		370,000	
Ultrafiltration system installation @ 40% of UF system cost		148,000	
Ultrafiltration system installed cost			518,000
Total equipment cost			725,340
Detailed engineering design @ 12% of auxiliary equipment installed cost and 12% of UF system installed cost			87,041
Building (186 m^2 @ $161/$m^2$) [2000 ft^2 @ $15/$ft^2$]			30,000
Subtotal A			842,381
Administration and supervision (2% of subtotal A)			16,848
Subtotal B			859,229
Contingency (10% of subtotal B)			85,922
Subtotal C			945,151
Inflation (10% of subtotal C)			94,515
Total installed cost			$ 1,039,666

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* Based on idealized spiral-wound modules. Not attainable with current technology.

TABLE 37. CASE 8 DESIGN CAPITAL COST SUMMARY*

Pretreatment system	155,700		
Permeate collection/ distribution subsystem	71,100		
Concentrate collection/ distribution subsystem	14,400		
		241,200	
Installation @ 40% of total auxiliary equipment cost		96,480	
Auxiliary equipment installed cost			337,680
Ultrafiltration system (includes $194,400 for membranes)		700,000	
Ultrafiltration system installation @ 40% of UF system cost		280,000	
Ultrafiltration system installed cost			980,000
Total equipment cost			1,317,680
Detailed engineering design @ 12% of auxiliary equipment installed cost and 12% of UF system installed cost			158,122
Building ($232\ \text{m}^2$ @ $\$161/\text{m}^2$) [$2{,}500\ \text{ft}^2$ @ $\$15/\text{ft}^2$]			37,500
Subtotal A			1,513,302
Administration and supervision (2% of subtotal A)			30,266
Subtotal B			1,543,568
Contingency (10% of subtotal B)			154,356
Subtotal C			1,697,924
Inflation (10% of subtotal C)			169,792
Total installed cost			$ 1,867,716

* Based on idealized spiral-wound modules. Not attainable with current technology.

TABLE 38. CASE 5 DESIGN OPERATING DATA*

Cost Element	Quantity	Unit Cost	$/day	$/year	$/m^3	$/M-gal	% of Total
Material (for cleaning)							
Caustic (50%)	37.6 kg/day	$ 0.154/kg	5.81	2,121			
EDTA	4.9 kg/day	1.95/kg	18.41	6,710			
Cartridge Filters (0.76 m)	16/day	4/cartridge	64.00	23,360			
Total materials			88.22	32,201	0.023	0.09	11.4
Conversion expense							
Labor (including benefits)	6 hr/day	$ 10/hr	60.00	21,900	0.016	0.06	7.6
Electrical power	2,237 kwh/day	$ 0.0025/kwh	50.30	18,360	0.013	0.05	6.3
Repair and Maintenance							
Material	1.5% of total equipmment less membrane replacement cost yearly = (1.5% x 673,540)/yr		27.68	10,103	0.007	0.03	3.8
Labor	2 hr/day	$ 15/hr	30.00	10,950	0.008	0.03	3.8
Insurance and Taxes	0.5 x maintenance material		13.84	5,052	0.004	0.01	1.3
Total, excluding depreciation			181.82	66,365	0.048	0.18	22.8
Depreciation							
Membranes	$ 121,800	3-yr life	333.70	121,800	0.088	0.33	41.8
Other facilities	$ 1,014,626	15-yr life	185.32	67,642	0.049	0.19	24.1
Total conversion expense			$ 700.84	255,807	0.185	0.70	88.6
Total incremental cost			$ 789.06	288,008	0.209	0.79	100.0

*Based on idealized spiral-wound modules. Not attainable with current technology.

TABLE 39. CASE 6 DESIGN OPERATING COST DATA*

Cost Element	Quantity	Unit Cost	$/day	$/year	$/m^3	$/M-gal	% of Total
Material (for cleaning)							
Caustic (50%)	53.8 kg/day	$ 0.154/kg	8.31	3,033			
EDTA	20.8 kg/day	1.95/kg	26.28	9,952			
Cartridge Filters (0.76 m)	32/day	$ 4/cartridge	128.00	46,720			
Total materials			162.59	59,345	0.022	0.08	11.6
Conversion expense							
Labor (including benefits)	6 hr/day	$ 10/hr	60.00	21,900	0.008	0.03	4.3
Electrical power	3,811 kwh/day	$ 0.0025/kwh	85.75	31,299	0.011	0.04	5.8
Repair and Maintenance							
Material	1.5% of total equipmment less membrane replacement cost yearly = (1.5% x 1,236,880)/yr		50.83	18,553	0.007	0.03	4.3
Labor	3.5 hr/day	$ 15/hr	52.50	19,163	0.007	0.03	4.3
Insurance and Taxes	0.5 x maintenance material		25.42	9,278	0.003	0.01	1.4
Total, excluding depreciation			274.50	100,193	0.036	0.14	20.3
Depreciation							
Membranes	$ 220,800	1-yr life	604.93	220,800	0.080	0.30	43.5
Other facilities	$ 1,840,439	15-yr life	336.15	122,696	0.044	0.17	24.6
Total conversion expense			$ 1,215.58	443,687	0.160	0.61	88.4
Total incremental cost			$ 1,378.17	503,032	0.182	0.69	100.0

* Based on idealized spiral-wound modules. Not attainable with current technology.

TABLE 40. CASE 7 DESIGN OPERATING COST DATA*

Cost Element	Quantity	Unit Cost	$/day	$/year	$/m^3	$/M-gal	% of Total
Material (for cleaning)							
Caustic (50%)	37.6 kg/day	$ 0.154/kg	5.81	2,121			
EDTA	4.9 kg/day	1.95/kg	18.41	6,720			
Cartridge Filters (0.76 m)	16/day	$ 4/cartridge	64.00	23,360			
Total materials			88.22	32,201	0.023	0.09	12.5
Conversion expense							
Labor (including benefits)	6 hr/day	$ 10/hr	60.00	21,900	0.016	0.06	8.3
Electrical power	1,915 kwh/day	$ 0.0025/kwh	40.09	14,633	0.011	0.04	5.6
Repair and Maintenance							
Material	1.5% of total equipmment less membrane replacement cost yearly = (1.5% x 617,940)/yr		25.39	9,269	0.007	0.03	4.2
Labor	2 hr/day	$ 15/hr	30.00	10,950	0.008	0.03	4.2
Insurance and Taxes	0.5 x maintenance material		12.70	4,636	0.003	0.01	1.4
Total, excluding depreciation			168.18	61,386	0.044	0.17	23.6
Depreciation							
Membranes	$ 107,400	1-yr life	294.25	107,400	0.078	0.29	40.3
Other facilities	$ 932,266	15-yr life	170.28	62,151	0.045	0.17	23.6
Total conversion expense			$ 632.71	203,939	0.167	0.63	87.5
Total incremental cost			$ 720.93	236,140	0.191	0.72	100.0

* Based on idealized spiral-wound modules. Not attainable with current technology.

TABLE 41. CASE 8 DESIGN OPERATING COST DATA*

Cost Element	Quantity	Unit Cost	$/day	$/year	$/m^3	$/M-gal	% of Total
Material (for cleaning)							
Caustic (50%)	53.8 kg/day	$ 0.154/kg	8.31	3,033			
EDTA	13.5 kg/day	1.95/kg	26.28	9,592			
Cartridge Filters (0.76 m)	32/day	$ 4/cartridge	128.00	46,720			
Total materials			162.59	59,345	0.021	0.08	12.7
Conversion expense							
Labor (including benefits)	6 hr/day	$ 10/hr	60.00	21,900	0.008	0.03	4.8
Electrical power	3,634 kwh/day	$ 0.0025/kwh	81.77	29,846	0.011	0.04	6.3
Repair and Maintenance							
Material	1.5% of total equipment less membrane replacement cost yearly = (1.5% x 1,123,280)/yr		46.16	16,849	0.006	0.02	3.2
Labor	3 hr/day	$ 15/hr	45.00	16,425	0.006	0.02	3.2
Insurance and Taxes	0.5 x maintenance material		23.08	8,424	0.003	0.01	1.6
Total, excluding depreciation			256.01	93,444	0.034	0.13	20.6
Depreciation							
Membranes	$ 194,400	1-yr life	532.60	194,400	0.070	0.27	42.9
Other facilities	$ 1,673,316	15-yr life	305.63	111,554	0.040	0.15	23.8
Total conversion expense			$ 1,094.24	399,398	0.144	0.55	87.3
Total incremental cost			$ 1,256.83	458,743	0.166	0.63	100.0

* Based on idealized spiral-wound modules. Not attainable with current technology.

TABLE 42. SUMMARY OF PROJECTED ECONOMICS FOR DESIGN CASES 5 THROUGH 8*

Item	Design Case Number			
	5	6	7	8
Stream	Caustic extraction filtrate	Caustic extraction filtrate	Decker effluent	Decker effluent
Flow, m^3/day (MM gal/day)	3,790(1)	7,580(2)	3,790(1)	7,580(2)
UF membrane cost, $	121,800	220,800	107,400	194,400
UF system cost (including membranes), $	420,000	800,000	370,000	700,000
Total installed cost, $	1,136,426	2,061,239	1,039,666	1,867,716
Daily operating cost, $	789	1,378	721	1,257
Cost per m^3, $	0.21	0.18	0.19	0.17
Cost per 1000 gal, $	0.79	0.69	0.72	0.63
Cost per metric ton of pulp, $	1.08	1.89	0.57	0.99
Cost per ton of pulp, $	0.99	1.72	0.50	0.87

* Based on idealized spiral-wound modules. Not attainable with current technology.

As observed in Table 42, capital investment ranges from $1 to $2.1 MM. This is roughly 50% of the installed costs for the tubular-based treatment systems. Treatment costs are $1.09 to $1.89/metric ton ($0.99 to $1.72/ton) of pulp for caustic extraction filtrate processing and $0.55 to $0.95/metric ton ($0.50 to $0.87/ton) of pulp for decker effluent processing.

POTENTIAL CREDITS FOR WATER REUSE AND RESOURCE RECOVERY

No credits have been applied to the treatment system operating costs for water reuse or resource recovery. A qualitative discussion of these potential credits is presented in the next section, entitled, Water Reuse Potential.

SECTION 9

WATER REUSE POTENTIAL

In the foregoing economic projections no credits have been applied to the treatment system operating costs for water (permeate) reuse or concentrate reuse. It is expected that for a mill installation these streams will be recycled to some extent and that operating cost credits will accrue. Reuse applications and potential credits are discussed below.

PINE BLEACHERY CAUSTIC EXTRACTION FILTRATE

The permeate from the ultrafiltration unit treating caustic extraction filtrate will constitute about 98% of the feed stream. This effluent will have low color, essentially no suspended solids and will have very low heavy metal content. In addition the permeate will have a high pH and be at process temperature.

The permeate, with its physical and chemical attributes, should be a superior water makeup stream for use in the bleachery processes. The high pH, and reduced buffering capacity should allow for lower new caustic requirements. The high temperature should reduce the system energy requirements. Because of the absence of suspended solids and the decreased heavy metal content the permeate should reduce spray head and other scaling problems.

No estimate is made here for the potential economic value of these factors since the potential will be quite site-specific. It is believed that at least half of the permeate can be used in a bleachery recycle mode and that the savings in chemicals, water and energy from such use will have substantial effects in reducing the net cost of the operation.

Permeate which is excess can be admixed with mill input fresh water without discernable effect upon the fresh water quality. This is because the small permeate volume would be diluted 25 to 50 times by the larger fresh water input.

Recycling all or substantially all of the permeate as above would have an additional benefit of reducing the flow of effluent to be treated in the waste disposal area by 2-4% which is not quantifiable in a generic case.

The concentrate from ultrafiltration has no direct value in pulp mill operations but it will be small in volume and because of its high organic solids content will be combustible. Disposal of this material would be site-specific. In those installations equipped to remove chlorides from black liquor systems, this concentrate could be flowed to the weak black liquor system with some small gain in energy recovery. Alternatively, the concentrate could be combusted in a typical modern lime kiln without noticeable effect, especially because of the low sodium content. Some mills which do not have sufficient lime kiln capacity dispose of the excess lime sludge off-site. Because of the high pH of this sludge and the relatively small volume of the concentrate, the concentrate could be added to the lime sludge and carried to land fill as insolubilized calcium salts.

Because the water reuse and resource recovery considerations for the ultrafiltration permeates and concentrates are site-specific no formal estimate of credits are projected here. It is felt that the reuse of permeate will result in values in reduction of water, chemical, energy and maintenance costs, and waste disposal system loading which will substantially reduce the net cost of operation of the ultrafiltration unit.

The disposal of the concentrate as discussed above should result in at least a break-even disposal cost.

PINE AND HARDWOOD PULP WASHING DECKER EFFLUENTS

In a previous study (32) it was demonstrated that ultrafiltration of decker effluents for the specific cases considered would reduce the net costs of color removal by ultrafiltration to break-even or even provide a return on the investment. Calculations based on the present tubular membrane economic projections indicate that recycling the permeates to the pulping system for makeup water and the addition of the concentrates to the weak black liquor system, although site-specific, still offer the potential for break even or low net cost operation of an ultrafiltration unit. The cost values here result from recovery and recycling of water, chemicals, energy and reduction of waste disposal system loading by 2-4%.

Large scale demonstrations of reuse or disposal were not possible in this study. The pilot quantities produced were small relative to the actual mill material flows. A substantially larger prototype demonstration plant could provide the quantities of permeates and concentrates required for reasonable scale reuse or disposal systems testing.

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APPENDICES

APPENDIX A. MEMBRANE CLEANING MATERIALS AND TECHNIQUES

Three classes of membrane cleaning materials were investigated:

- detergents
- enzymes
- chelating agents

The following materials and combinations were investigated for membrane cleaning effectiveness.

a. Ultraclean (Abcor)
b. Enzyme (Abcor)
c. Iron Chelating Agent (Abcor)
d. C-S Detergent (Osmonics)
e. Ultrazme (Osmonics)
f. Caustic and EDTA
g. Oxalic acid
h. Ultraclean and caustic
i. Ultraclean and EDTA
j. Ultraclean and Iron chelating agent
k. Ultraclean and Enzyme

During the membrane cleaning studies sufficient work was done with the chelating agents to determine that the "fouled" membrane was not the result of metal fouling.

Plugging of the spiral wound membrane modules was not a problem during this program. The prefiltration system removed the suspended particles and cleaned the stream sufficiently to prevent plugging. A "dirty" module was the result of a formed boundary layer on the membrane surface that needed to be "scoured" from the surface. Backflushing of the module was not at all effective in cleaning a fouled membrane. Soaking of the module in water or sufficient recirculation time with a cleaning solution to loosen the bound foulant layer proved the only effective means to clean the modules.

APPENDIX B. DERIVATION OF THE EQUATION RELATING INTRINSIC REJECTION TO APPARENT REJECTION

The intrinsic rejection (R_i) is defined as follows:

$$R_i = \frac{C_f - C_p}{C_f} = 1 - \frac{C_p}{C_f} \qquad (1)$$

where

C_p = Concentration in the permeate
C_f = Concentration in the feed

The apparent rejection (R_a) is defined as follows:

$$R_a = \frac{C_{f_o} - \overline{C}_p}{C_f} = 1 - \frac{\overline{C}_p}{C_f} \qquad (2)$$

where

$\overline{C}_p$ = Average concentration in the permeate
C_{f_o} = Original concentration in the feed (assumed equal to C_f in calculating R_a)

Equation 1 can be rearranged to yield

$$C_p = (1-R_i)C_f \qquad (3)$$

The concentration in the feed as a function of volume processed can be expressed as follows:

$$C_f = C_{f_o} \left(\frac{Vo}{V}\right)^{-R_i} \qquad (4)$$

where

Vo = Initial volume in the cell
V = Volume in the cell at the time t

V and Vo can be related to the conversion (Y) as follows:

$$Y = \frac{Vo - V}{Vo} = 1 - \frac{V}{Vo} \qquad (5)$$

Equation 5 can then be rearranged and substituted into equation 4 to yield:

$$C_f = C_{f_o} \left(1 - Y\right)^{-R_i} \qquad (6)$$

Equation 6 can then be substituted into equation 3 to yield:

$$C_p = (1 - R_i)C_{f_o} (1 - Y)^{-R_i}$$

or

$$\frac{C_p}{C_{f_o}} = (1 - R_i)(1 - Y)^{-R_i} \qquad (7)$$

Integration of equation 7 with respect to Y yields:

$$\int_{Y_1}^{Y_2} \frac{C_p}{C_{f_o}} \, dy = (1 - R_i) \int_{Y_1}^{Y_2} (1 - Y)^{-R_i} \, dy$$

or

$$\frac{\overline{C}_p}{C_{f_o}} = \frac{(1 - Y_1)^{1-R_i} - (1 - Y_2)^{1-R_i}}{(Y_2 - Y_1)}. \qquad (8)$$

Substituting equation 2 into equation 8 yields:

$$R_a = 1 - \left[\frac{(1 - Y_1)^{1-R_i} - (1 - Y_2)^{1-R_i}}{Y_2 - Y_1}\right]$$

APPENDIX C. ADDITIONAL DATA FROM MEMBRANE SELECTION STUDIES

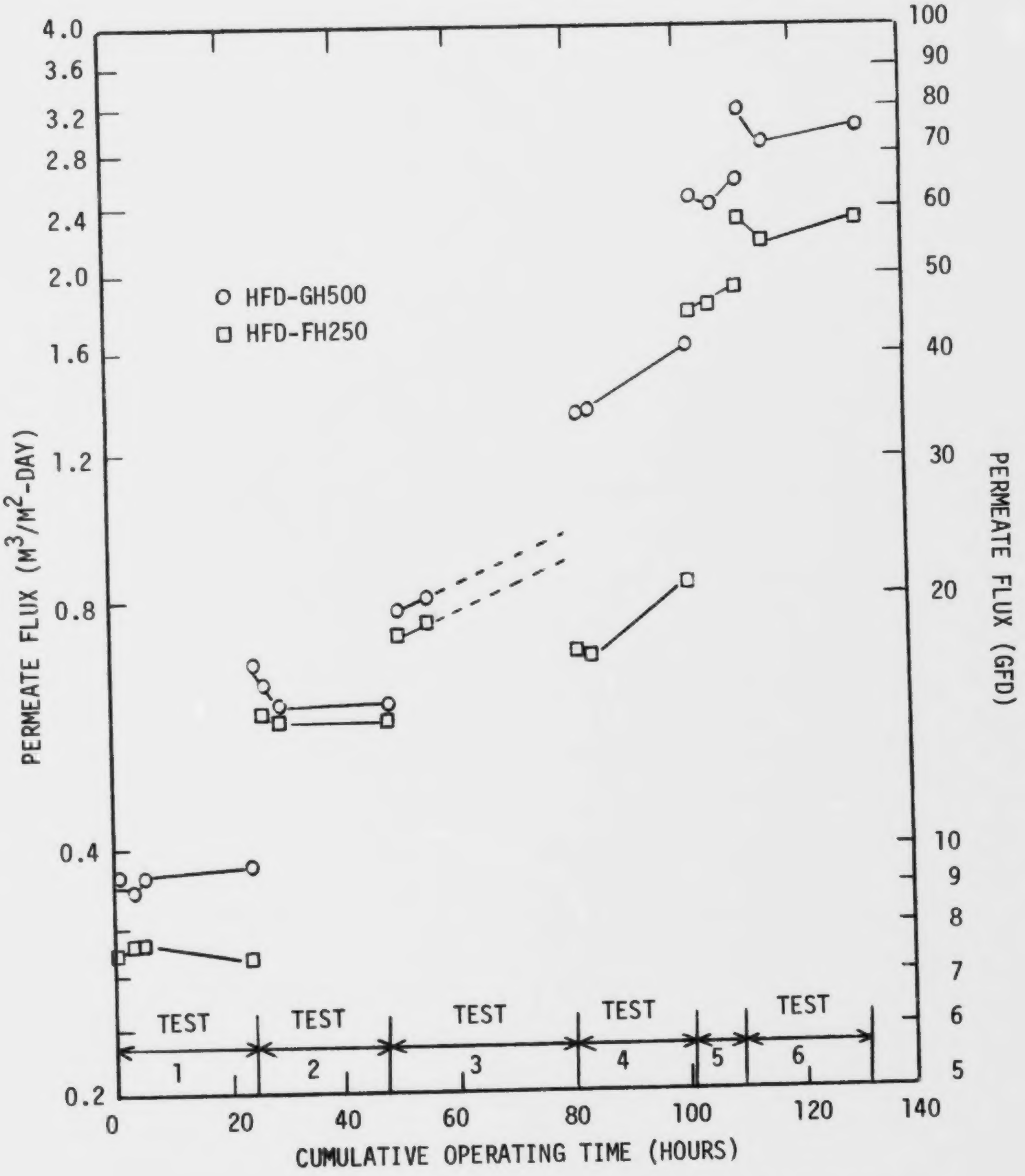
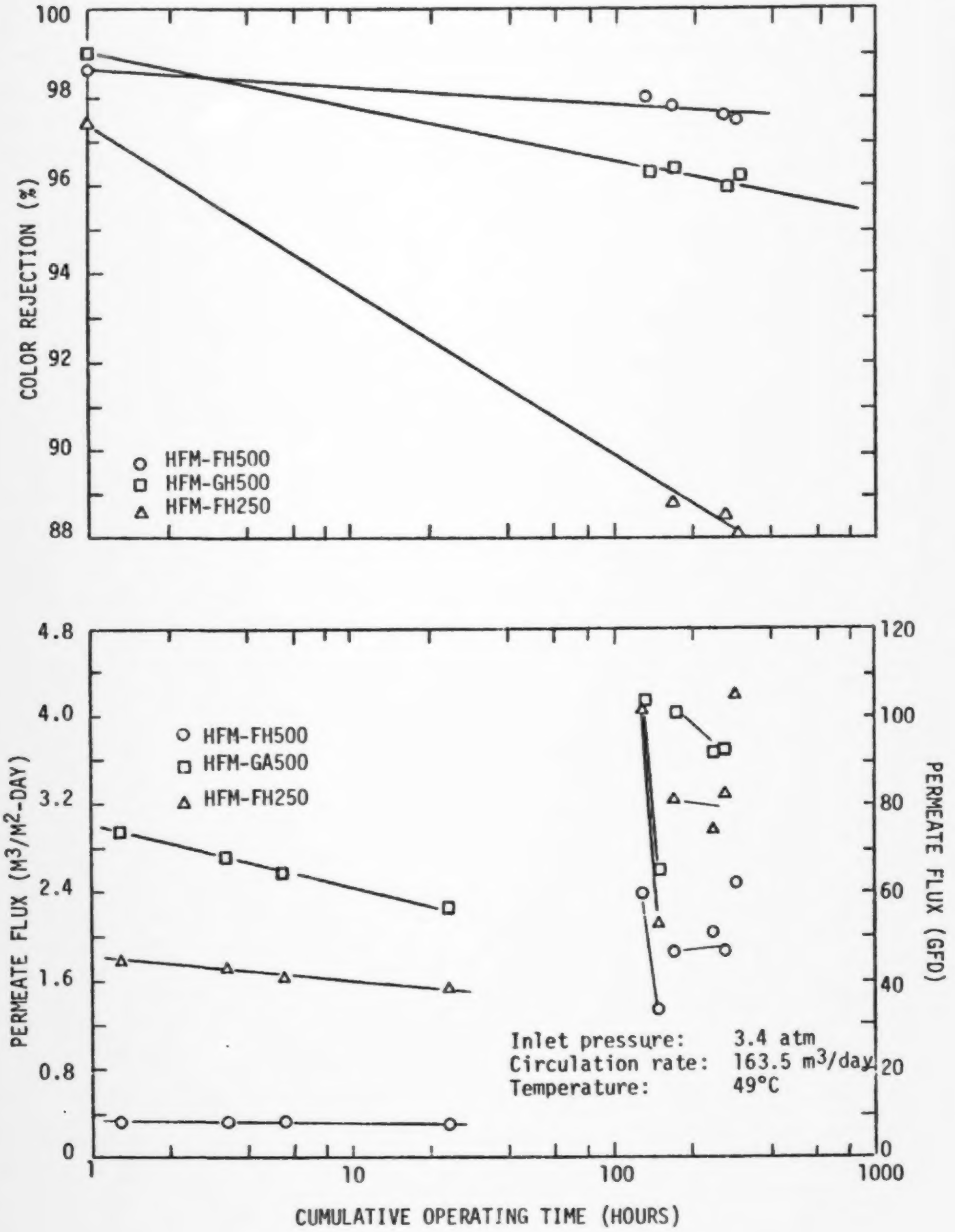


Figure C1. Coated HFD membrane flux data obtained during parametric studies.



Inlet pressure: 3.4 atm
Circulation rate: 163.5 m^3/day
Temperature: 49°C

Figure C2. Coated HFM membrane flux and rejection characteristics at constant operating conditions.

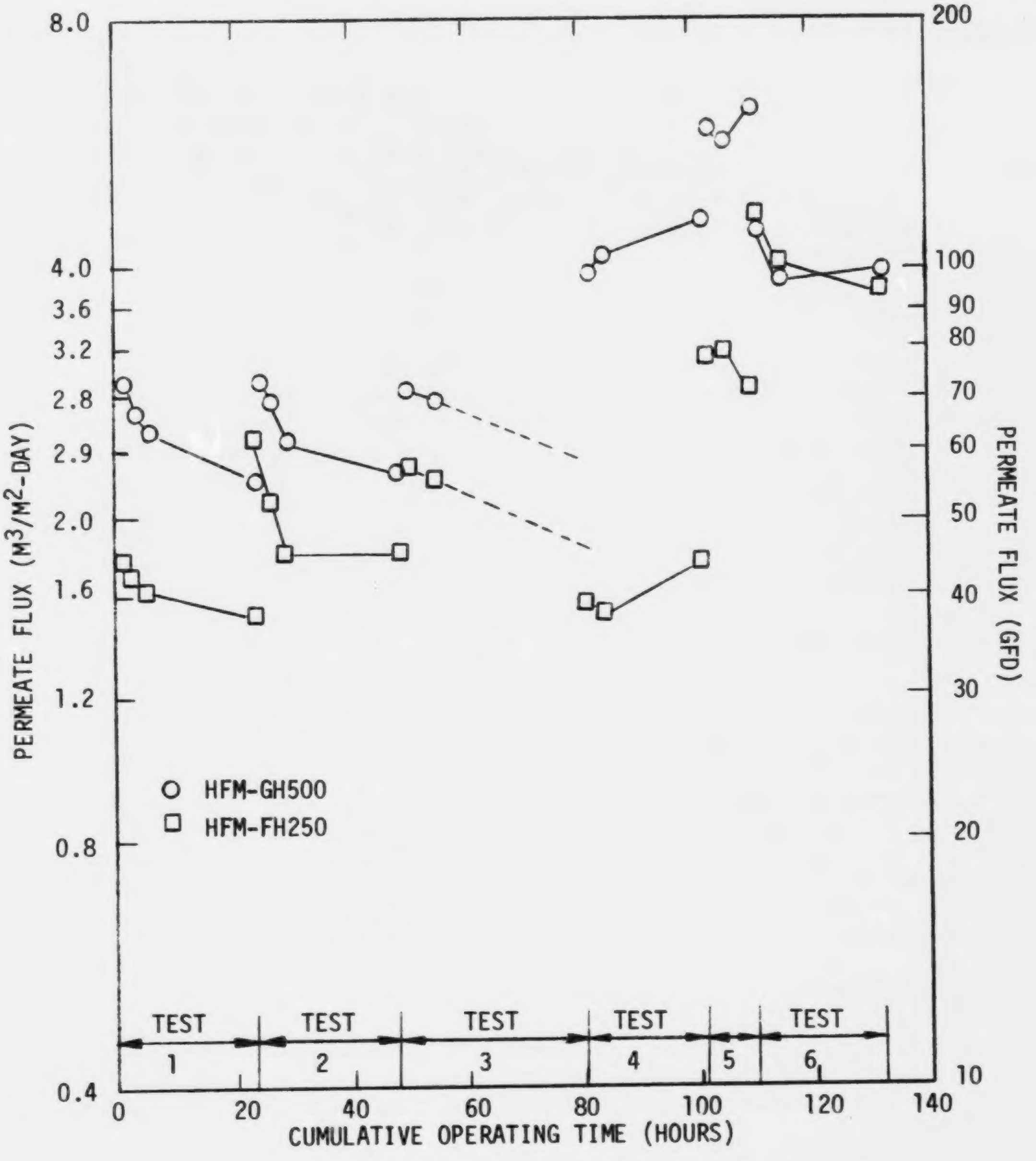


Figure C3. Coated HFM membrane flux data obtained during parametric studies.

APPENDIX D. FINAL REPORT - USE OF SULFONIC ACID MEMBRANES FOR TREATMENT OF PULP AND PAPER WASTE STREAM - PREPARED BY H. GREGOR, COLUMBIA UNIVERSITY, SUBCONTRACT TO CHAMPION, P.O. 165723*

PURPOSE

The purpose of this subcontract was to have Gregor, on the basis of his laboratory tests, select sulfonic acid membranes which appeared to be suitable for the treatment of feeds supplied by Champion, using analytical procedures prescribed by Champion. Selected samples were to be supplied to Champion for further laboratory tests by them, and then Champion was to select membranes to be supplied in 0.3 m x 1.8 m (1 ft x 6 ft) sizes for pilot plant studies.

MEMBRANES AND SUPPORTS

The membranes of US Patent 3,808,305 can be cast on a variety of support materials. Those which had been employed were examined and a support membrane which was believed to be well suited for the Champion study was selected. Its chemical stability was evaluated according to specifications given by Dr. Fremont (Champion), by subjecting it to solutions at pH 12 (with sodium carbonate) at 57°C (135°F) for several weeks. It was found that the material was quite stable under these conditions. Then the temperature was increased to 90°C, much more extreme than that encountered in field use, and the support membranes continued to be stable.

Membranes Employed

All of the membranes employed for this study were made in accordance with US Patent 3,808,305, and consisted primarily of sulfonic acid polymers to make them non-fouling, and were cured by chemical cross-linking. They were made in a variety of porosities and were characterized by measuring their hydraulic permeabilities or fluxes, usually at room temperature in water and in the presence of the dye erythrocin at the 15 ppm level. Membranes are characterized according to their hydraulic permeabilities in microns per second - atmosphere (μsa) by their dye rejections as percent rejection in the usual manner. Membranes of selected porosity and other characteristics were employed for this study.

TEST EQUIPMENT

In order to meet the specifications set down for this study, the cells employed were of the Gelman stainless steel variety as has been described in the original proposal to Champion. These have a small stainless steel insert screwed into the upper plate of the cells so as to allow for the control of convection across the face of the membrane as feed solution is pumped through the insert, across the face of the membrane and out the exit port. The distance of separation from the insert to the membrane surface was 0.5 mm and the radius of average velocity was readily calculated to be 0.822 centimeters for the insert of this dimension. Thus, the area of flow at the radius of average velocity was 0.26 cm^2. These cells have an effective membrane area of 13.5 cm^2, so a 2 μsa membrane at 50 psi would provide for a flux of about 30 ml per hour. These cells are shown in Figure D-1.

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* This final report is reproduced exactly as received except for required format changes (retyping, etc.).

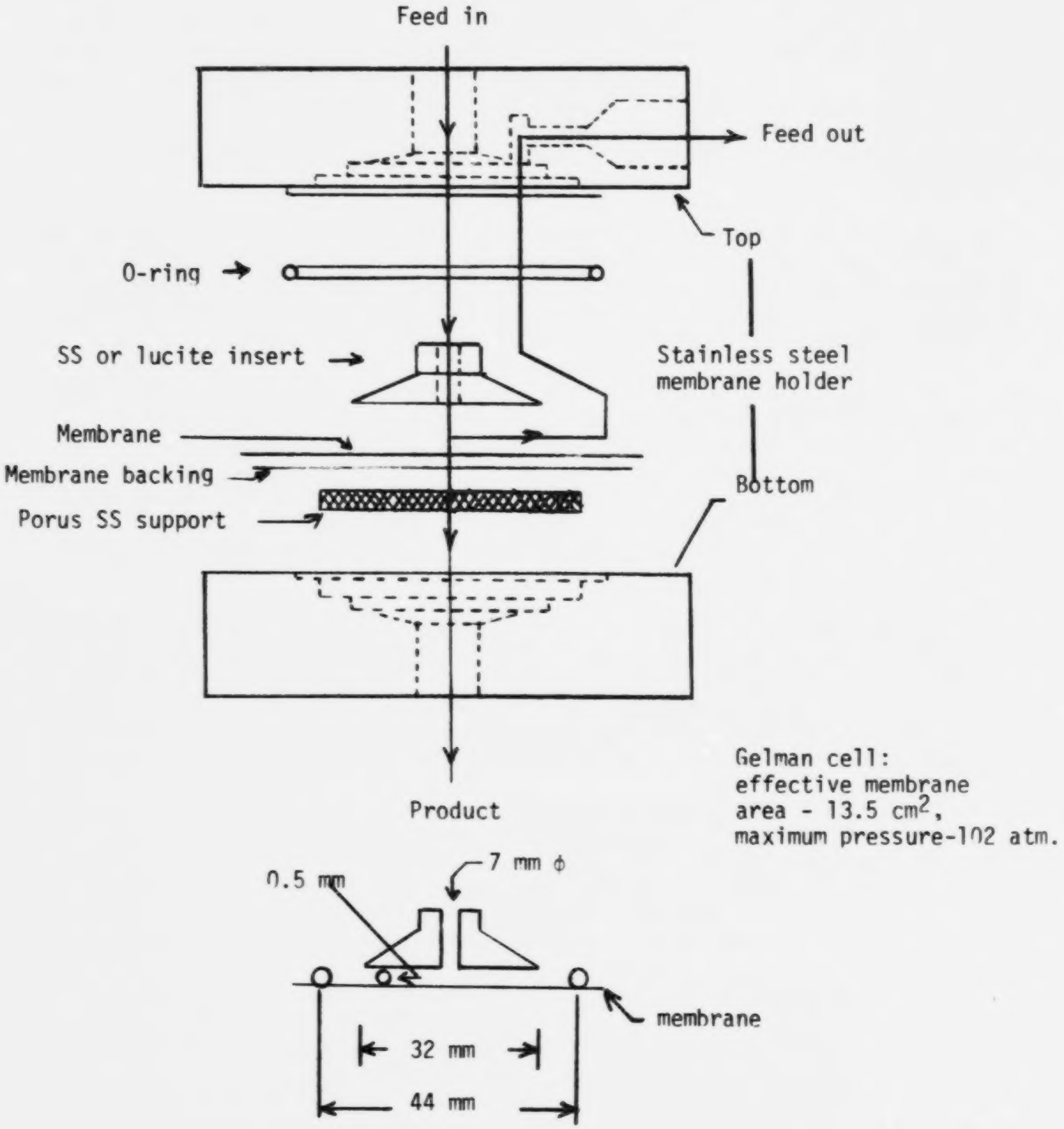
The rate of feed recirculation across the face of the membrane was set by the voltage supplied to the circulating pump which operated quite independently of the total pressure imposed on the system. Tests showed that average circulation velocities of greater than 0.5 meter/sec did not lead to higher fluxes, so a rate of 0.5 meter/sec was maintained.

These cells were mounted into a batch-type unit (see Figure D-2) constructed entirely of stainless steel, having a reservoir volume of approximately 2.5 liters, a circulating pump allowing for a rate of convection across the membrane face which could be varied at will over wide ranges, capable of operation at temperatures well above 100°C and at quite high pressures. The residual volume of this device was 60 ml, so in a single run at that volume of feed the device could be operated to approximately 94% water recovery.

The absorbance of feeds, concentrates and permeates was determined as follows, as specified by Champion. First, the pH was measured and then the sample was diluted with distilled water until the absorbance reached the useful range of the instrument, which was either a Spectronic 20 or Beckman DU at 465 nm. At this point the pH was adjusted with sulfuric acid to 7.6 and the final reading was made of the original absorbance of that solution.

The mixed permeate assays were also determined directly by collecting all of the permeate at a given degree of water recovery and subjecting this collected permeate to the same analysis and reporting the results in that manner. One can readily calculate the absorbance of the mixed permeate and concentrate by calculations from the point by point composition of the permeate. This was not done because Abcor preferred the direct determination.

The specifications for this project were ultimately defined by Dr. Gollan (Abcor): All runs were to be made at 140°F or 60°C. As regards fluxes measured at 5.1 atm (75 psi), a membrane would be considered "poor" if its flux were less than 0.83 m^3/m^2-day (20 gfd) and the very good membrane would have a flux greater than 3.28 m^3/m^2-day (80 gfd). With respect to rejection, on a mixed permeate up to 98% water recovery, a rejection of greater than 90% was considered desirable.


Feed in
Feed out
Top
O-ring
SS or lucite insert
Stainless steel
membrane holder
Membrane
Membrane backing
Bottom
Porus SS support
Product
0.5 mm
7 mm ϕ
membrane
32 mm
44 mm

Gelman cell:
effective membrane
area - 13.5 cm^2,
maximum pressure-102 atm.

Figure D1. Modified Gelman filter holder.

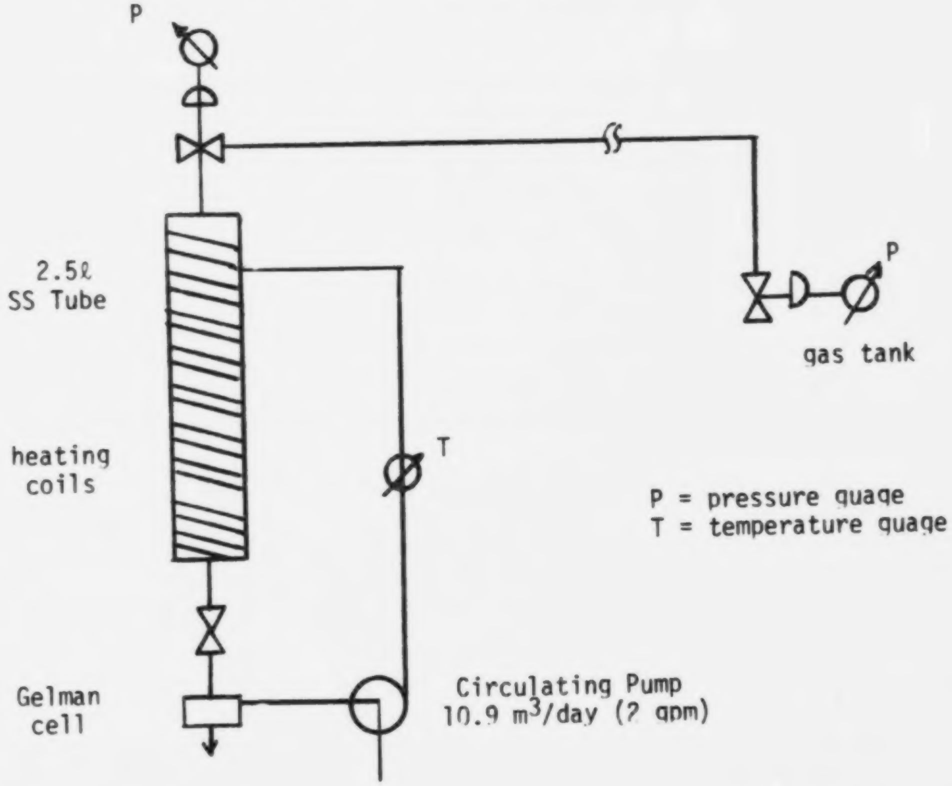

P
2.5ℓ
SS Tube
heating
coils
Gelman
cell
T
Circulating Pump
10.9 m3/day (2 gpm)
P
gas tank
P = pressure guage
T = temperature guage

Figure D2. High pressure-high temperature UF/RO assembly.

The specifications of this project were subject to some variation as the management of the project changed.

MEMBRANE SAMPLES SUBMITTED

A number of membrane samples were submitted. The set labeled A-E had the following characteristics:

TABLE D-1. MEMBRANE PROPERTIES

Designation	J_{sa}	%DR
A	10	<10
B	5.0	25
C	2.6	68
D	0.80	97
E	1.3	80

The membrane types used in the runs of Tables D2-D5 are designated (A - E) by membrane type. A number of different variants within each type were prepared and tested; the variant was usually that of thickness. For example, membranes of designation D all had high rejections of dye, and making them in thinner forms gave films of higher flux, with little loss in dye rejection.

RESULTS OBTAINED - DISCUSSION

A number of membranes were subjected to tests at 80°C at 6.8 atm (100 psi). Membranes having a wide range of dye rejections from nearly zero to 99% were tested; these had fluxes from 0.5 μsa as high as 30 μsa, all in water at room temperature. Only in those cases when membranes gave a reasonable flux and a good level of dye rejection was the run continued to the end. A number of preliminary experiments were also performed with the Champion feed at room temperature, but here it was found that the results were not at all indicative of those obtained at higher temperatures. At general, fluxes were lower as expected, but the color rejection in some cases was erratic, possibly due to association of lower molecular weight color bodies in the feed with other molecules of higher molecular weight which never permeated the membrane. The color of the feed stayed constant over the entire period of its use.

A few experiments were also performed to ascertain the performance of the membrane operating at different temperatures because of the change in the specifications from 80°C to 60°C. It was found that above 50°C on the Champion feed the membranes of our study gave rather similar fluxes and dye rejections, with a 10% to 20% increase in the ultrafiltration rate at 80°C as compared with 60°C. However, at that point the project was terminated so no definitive tests were performed thereafter.

Tables D2 to D6 summarize the results obtained. Table D6 shows that rejection in all cases was well above specifications, that the flux in one case was at the minimal level, in others at the good level and in two at the very good level, being well above 3.28 m^3/m^2-day (80 gfd) at 5.1 atm (75 psi).

The pH of the permeate (Table D2) was slightly higher than of the feed, as is usually the case when material which is a salt of a polyacid is ultrafiltered. A small degree of hydrolysis of the salt results because the counterions (Na^+) are forced through the membrane while the polyanion remains in the feed. Electroneutrality is maintained by hydrolysis, producing NaOH in the permeate and the acid in the feed. These kinds of pH shifts are frequently found in treating natural feeds.

In Table D3, the pH of the permeate was less than that of the feed, and this difference increased throughout the run. One could postulate that polycations were present, but this is most unlikely at these high pH levels. Table D4 shows that the feed was 2 pH units above the first permeate, and this difference persisted. Table D5 also shows a much lower pH for the permeate than for the feed. At the 25%, 50% and 75% treatment points, the permeate was at pH 9.2 and the concentrate at 8.7-8.9, or the normal increase in pH was found.

The explanation which suggests itself is that there are colloidal aggregates present in the original feed which impart a higher pH (by adsorption to the electrode, possibly), but that these settle out as the run continues where the expected pH difference between feed and permeate is observed. Concentrate compositions and pH levels were always taken from the circulating feed stream, so if material settled out at the bottom of the feed tank where it would not appear in the concentrate, the pH shifts observed would result.

It was not possible for us to evaluate or discuss the fundamental aspects of the results obtained because no information on the chemical composition of the feeds was supplied. Two feed samples were supplied. The first had a pH of 8.5, it was small in volume 3.785 liters [1 gal] and was consumed in a series of preliminary runs. The second sample was larger and had a pH of 10.3. The former sample was darker, with an absorbance of 5.460, while the latter sample had an absorbance of 4.375 ± 0.010. Both

TABLE D-2. CHAMPION EFFLUENT - RUN #12

Soln	pH	Abs	%Rej	% Water Rec	Flux, m^3/m^2-day (gfd)
Feed	8.5	5.460	-	-	-
P1	8.8	.104	98	6	1.23 (30)
P2	8.9	.154	97	16	1.31 (32)
P3	8.6	.216	96	25	1.31 (32)
C3	8.5	9.50	-	25	
P4	8.6	.268	95	34	1.31 (32)
C4	8.5	10.6	-	34	
P5	8.9	.328	94	43	1.68 (41)
C5	8.8	12.6	-	43	
P6	8.9	.348	94	53	1.56 (38)
C6	8.8	12.6	-	53	
P7	8.9	.364	93	59	1.52 (37)
C7	8.8	13.1	-	59	
P8	8.9	.324	93	70	1.56 (38)

P - Permeate
C - Concentrate

Feed: 1000 ml T : 80°C, 176°F
Cell: SS, 13.5 cm^2 P: 100 psi
Memb: No. 27, 1.2 μsa, 93% DR (Series D)

TABLE D-3. CHAMPION EFFLUENT - RUN #16

Soln	pH	Abs	%Rej	% Water Rec	Flux, m^3/m^2-day (gfd)
Feed	10.3	4.375	-	-	-
P1	9.5	.727	85	11.0	6.8 (167)
P2	9.5	.428	90	21.0	6.8 (167)
P3	9.4	.321	93	33.5	6.8 (167)
C3		4.51			
P4	9.3	.253	94	45.0	6.8 (167)
C4		4.57	-	34	
P5	9.2	.219	95	55.3	6.8 (167)
C5		5.28	-	43	
P6	9.3	.222	97	73.1	6.8 (167)
C6		7.71	-	53	
P7	9.0	.327	97	83.1	6.8 (167)
C7		10.25	-	59	
P8	8.9	.410	97	93.1	6.86 (167)
C8		13.48			
P9	8.5	1.510	96	98.0	3.5 (85)
C9		36.95			

P - Permeate
C - Concentrate

Feed: 1000 ml T : 80°C, 176°F
Cell: SS, 13.5 cm^2 P: 100 psi
Memb: No. 32, 1.5 µsa, 78% DR (Series E)

TABLE D-4. CHAMPION EFFLUENT - RUN #17

Soln	pH	Abs	%Rej	% Water Rec	Flux, m^3/m^2-day (gfd)
Feed	10.3	4.375	-	-	-
P1	8.3	.349	92	11.5	5.1 (125)
P2	8.4	.316	93	21.5	5.1 (125)
P3	8.5	.310	93	32.7	5.1 (125)
P4	8.5	.364	96	43.3	5.1 (125)
C4	-	9.50	-	43.3	
P5	8.4	.400	97	60.2	5.1 (125)
C5	-	12.75	-	60.2	
P6	8.7	.541	96	70.7	5.1 (125)
C6	-	15.60	-	70.7	
P7	8.6	.820	97	80.7	5.1 (125)
C7	-	25.72	-	80.7	
P8	8.5	5.320	93	90.7	3.0 (73)
C8	-	74.00	-	90.7	

P - Permeate
C - Concentrate
* Abs is measured for appropriately diluted solutions at pH 7.6 at 465 nm, and reported as the calculated abs of that solution.

Feed: 1000 ml T : 80°C, 176°F
Cell: SS, 13.5 cm^2 P: 100 psi
Memb: No. 31, 5.0 μ sa, 99% DR (Series E)

TABLE D-5. CHAMPION EFFLUENT - RUN #18

Soln	pH	Abs	%Rej	% Water Rec	Flux, m^3/m^2-day (gfd)
Feed	10.3	4.05	-	-	-
P1	9.2	.051	99	10.5	3.6 (87)
P2	9.2	.063	99	20.5	3.6 (87)
C2		4.30			
P3	9.2	.073	98	25.5	3.6 (87)
C3		4.50			
MP-25%	9.2	.063	98	25	3.6 (87)
Conc-25%	8.9	4.28		25	3.6 (87)
P4	9.3	.077	99	35.7	3.6 (87)
C4		5.30			
P5	9.5	.092	99	45.7	3.6 (87)
C5		6.40			
P6	9.2	.128	98	50.7	3.6 (87)
C6		6.95			
MP-50%	9.2	.098	98	50	3.6 (87)
Conc-50%	8.8	5.21		50	3.6 (87)
P7	9.2	.150	98	60.7	7.6 (87)
C7		9.60			
P8	9.2	.190	98	70.7	3.6 (87)
C8		12.35			
P9	9.0	.311	98	75.7	3.6 (87)
C9		16.55			
MP-75%	9.2	.217	98	75	3.6 (87)
Conc-75%	8.8	7.75		75	3.6 (87)
P10	9.0	.076	99	85.7	3.6 (87)
C10		66.50			
P11	9.0	2.88	97	90.0	3.6 (50)
C11		84.00			
MP-90%	-	1.82	98	90	
Conc-90%	-	24.5		90	

P - Permeate
C - Concentrate
MP - % means mixed permeate collected to that % water recovery.

Feed: 1000 ml
Cell: SS, 13.5 cm^2
Memb: No. 39, 8.0 μsa, 99% CR (Series E)
T : 80°C, 176°F
P: 100 psi

TABLE D-6. SUMMARY OF RUNS

No	T in 0°C	PSI*	J-100 psi*	J-75 psi*	CR
12	80	100	34	26	94
16	80	100	167	125	96
17	80	100	125	94	93
18	60	100	87	65	98.5

J-75 values were calculated from J-100 data.
* 75 psi - 5.1 atm; 100 psi - 6.8 atm.

samples were stored in the refrigerator, allowed to come to room temperature, shaken vigorously and then decanted rapidly into the test cell. The pH of these two feeds did not change over the period of use, and there was no evidence of flocculation or settling during the period of our tests. No pretreatment was ever employed; the feed material as supplied was used directly. There were no identification numbers of the samples as received, other than the designation Dan C. Tate, 2-14-087.

APPENDIX E. PRETREATMENT STUDIES

POLYMER PRETREATMENT OF FEED STREAMS

The rapid flux decline of spiral wound WRP membrane modules was a major problem. The analysis of the foulants from the surface of a fouled membrane gave the following analytical results (see attached Nalco analysis).

Volatile at 105°C	-88.0%
Kaolinite Clay	-5.6%
Starch	-4.0%
Titanium Dixoide	-1.4%
Carboxylic Acid Salt	-<1.0%

These data show that the major components in the "slime" layer come from recycle of white water from the paper mill back to the pulp mill. The prefiltration filters were effective in removal of the large suspended particles that would plug a module. However, the very small particles were getting into the membrane module and contributing to the fouling problem.

A study was made to find a polymer treatment that would flocculate these small suspended particles and enable the pre-filters to remove them.

Jar tests showed the following Nalco coagulants had the given order of activity at 3000 ppm dosage: GWP-827 > 7132 ≥ 107 ≥ 8103.

Besides the Nalco cationic coagulants, the following materials were investigated with jar studies: Chitosan, Arquad 2HT75, animal glue, lime, and acid addition with the Nalco polymers.

Based on the results of the jar studies, the Nalco polymers were investigated for efficacy with a 51 mm (2 in) diameter deep bed laboratory filter. The results of these experiments was at best inconclusive. The polymers form a gelatinous floc that is not completely removed by the deep bed filter or else forms (or reforms) after this filter. This results in high suspended solids test data. Results are in Table E1.

In the jar tests, addition of polymer resulted in substantial color removal when the floc settled. There was no evidence of this color removal with the laboratory deep bed filter studies.

A full scale trial was run with the Nalco 107 cationic polymer. During the trial, Nalco 7763 was added to aid the floc formation.

A flow schematic for the Nalco 107 polymer trial is shown in Figure E1. The operating procedure was as follows:

TABLE E-1

RESULTS OF 2" DEEP BED LABORATORY FILTER WITH CALCo POLYMERS AND CAUSTIC EXTRACT FILTRATE

	Polymer Conc	Feed				Filtrate			
		T.S.	D.S.	S.S.	Color	T.S.	D.S.	S.S.	Color
Nalco 107	3000 ppm	6752	6656	72	19,331	6840	6752	48	19,998
Nalco 107	3000 ppm	7248	7184	68	21,298	7260	7104	36	21,298
Nalco 107	1000 ppm	5824	5784	32	17,998	5196	5128	12	15,998
Nalco 6WP 827	500 ppm	6444	6368	42	18,332	6488	6368	100	18,665
Nalco 6WP 827/677	500/8 ppm	6350	6172	32	16,832	6216	6140	60	17,332
Nalco 6WP 827/677	300/8 ppm	7376	7296	36	15,998	7376	7308	36	16,332
Nalco 8103	3000 ppm	6816	6680	88	28,665	6788	6500	224	27,665
Nalco 8103	1000 ppm	7280	7172	64	21,331	7260	7072	152	21,431
Nalco 8103/677	500/8 ppm	7316	7232	44	19,265	7244	7124	80	19,398
Nalco 107/677	500/8 ppm	5436	5408	32	15,832	5420	5412	12	15,832

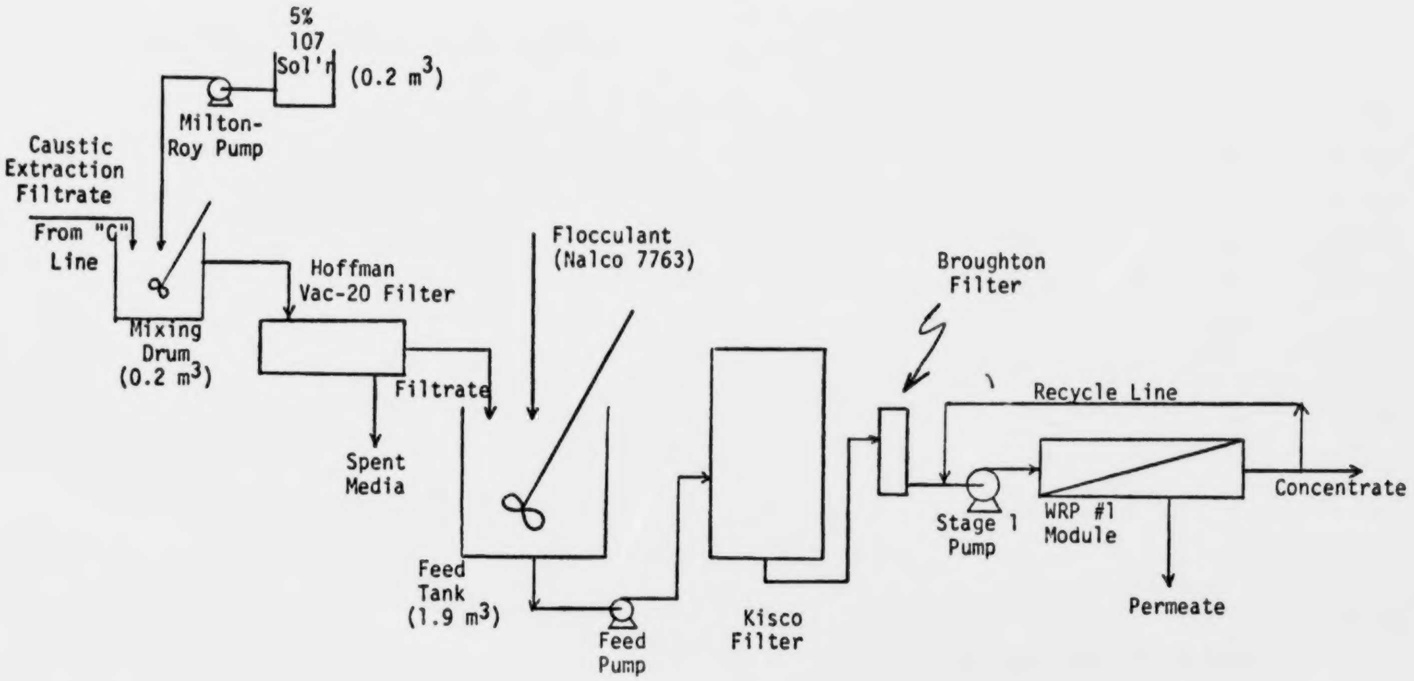

5%
107
Sol'n
(0.2 m^3)
Milton-
Roy Pump
Caustic
Extraction
Filtrate
From "C"
Line
Mixing
Drum
(0.2 m^3)
Hoffman
Vac-20 Filter
Filtrate
Spent
Media
Flocculant
(Nalco 7763)
Feed
Tank
(1.9 m^3)
Feed
Pump
Kisco
Filter
Broughton
Filter
Recycle Line
Stage 1
Pump
WRP #1
Module
Concentrate
Permeate

Figure E1. Flow schematic for Nalco 107 polymer trial.

- A 5% solution of Nalco 107 was made in the 0.21 m^3 (55 gal) polymer solution supply drum. The 107 polymer solution was pumped from the drum with a Milton-Roy positive displacement pump. The pump was calibrated and the flow setting adjusted to supply the quantity of 5% polymer solution necessary to give 3000 ppm polymer based on the flow of caustic extraction filtrate to the mixing drum.

- The flow of caustic extraction filtrate to the mixing drum was adjusted by valve to match the flow of filtrate from the Hoffman Vac-20 to the 1.9 m^3 (500 gal) feed tank. The Vac-20 has a built-in pump to pump filtrate. This flow was 21.8 m^3/day (4 gpm) during the trial. There was a stirrer on the mixing drum with a 2-inch diameter propeller rotating at 200 rpm. The level in the mixing drum was kept between 0.11 and 0.15 m^3 (30 and 40 gal) for the trial.

- The mixture of caustic extraction filtrate/polymer was gravity fed from the mixing drum to the Hoffman Vac-20 filter. The pool volume on the filter varied as the filter media was "blinded" and fresh filter media indexed. An estimate of variation in pool volume was from 0.004 to 0.02 m^3 (1 to 5 gal). The supply pipe from mixing drum to filter was 51 mm (2 in) in diameter and 6.1 m (20 ft) long. This contained less than 0.015 m^3 (4 gal) of liquid.

- The filtrate from the Vac-20 was pumped to the 1.9 m^3 (500 gal) feed tank. The flow from the feed tank through the test of the system was the normal system flow.

- A flocculant, Nalco 7763, was added to the feed tank during the last part of the trial. Once it was obvious the Nalco 107 coagulant was not solving the foulant problem, 8 ppm of Nalco 7763 was added to the system. This level of addition was sufficient during jar tests to aid the floc formation. The Nalco 7763 was added as a 5% solution. An initial addition sufficient to give 8 ppm in the 1.9 m^3 (500 gal) feed tank was made. Subsequent additions were made every thirty minutes to maintain the 8 ppm level.

- The stage 1 shell of the pilot system was used to hold the WRP #1 module for the trial since the single module test stand has no recycle capability. Because the through-put of the Hoffman Vac-20 filter was 21.8 m^3/day (4 gpm), this was the maximum rate of fresh feed to the UF system. The trial was run with 21.8 m^3/day (4 gpm) fresh feed and a 54.5 m^3/day (10 gpm) recycle rate for a total flow rate of 6.3 m^3/day (14 gpm) through the membrane module.

The results of the Nalco 107 polymer trial were very discouraging. While the Nalco 107 does cause coagulation of the suspended matter in the caustic extraction filtrate, the floc formed has no tenacity. The least bit of agitation causes the floc to break and pass through filters in the prefiltration system. The floc reforms when the stream comes to rest.

The data show extremely high suspended solids for samples taken after the Hoffman Vac-20 and the Broughton filters. The samples when taken looked very clear. The one-half hour time prior to testing was sufficient for copious amounts of floc to form. Table E1 shows the pH, suspended solids, total solids and color data taken during the trial.

The suspended solids data in Table E1 show the poor system operation. The feed to the system was around 100 ppm suspended solids. Yet, values from 1000-2800 ppm suspended solids were found after polymer addition and filtration through the Vac-20, Kisco, and Broughton filters.

Prior to the Hoffman Vac-20 filter, the system was only mildly mixed. The mixing drum was very lightly stirred and flow from the mixing drum to the Vac-20 was by gravity. However, the volumes involved were small so the system was never at rest. The Kisco deep bed filters have a liquid volume of 0.57 m^3 (150 gal) above the bed. At a flow rate of 21.8 m^3/day (4 gpm), there would be an average residence time of 40 minutes while going through the Kisco filters. This should be sufficient to allow the floc to reform and be filtered from the system. The data for suspended solids show the floc reforming in samples taken after the Broughton filters. Therefore, most of the floc is making it through the Kisco filters.

The loose floc problem was discussed with the Nalco people and when told that the Nalco 7763 flocculant was tried during the trial, could offer no suggestions for improvement.

Based on the experience gained during the polymer trial, the only hope for polymer addition to remove foulants would be the use of large settling tanks for floc removal. The ultrafiltration pilot area does not have this capability. Also, this would not be a viable solution for a full-scale unit.

The addition of Nalco 107 polymer had an adverse effect on flux rate during the trials. The breaking of the floc, which then passed through the total system, was the cause. Unless complete floc removal can be obtained, polymer addition only compounds the flux decline problem. The data in Table E3 show the rate of flux decline.

Figure E2 shows a comparison of the data in Table E3 versys flux decline without Nalco 107 polymer. The loss of flux occurring faster when the Nalco 107 was used.

TABLE E1. RESULTS OF 51 MM DIAMETER DEEP BED LABORATORY FILTER WITH NALCO POLYMERS AND CAUSTIC EXTRACTION FILTRATE

	Polymer concentration	Feed				Filtrate			
		T.S.	D.S.	S.S.	Color	T.S.	D.S.	S.S.	Color
Nalco 107	3000 ppm	6752	6656	72	19,331	6840	6752	48	19,998
Nalco 107	3000 ppm	7248	7184	68	21,298	7260	7104	36	21,298
Nalco 107	1000 ppm	5824	5784	32	17,998	5196	5128	12	15,998
Nalco GWP 827	500 ppm	6444	6368	42	18,332	6488	6368	100	18,665
Nalco GWP 827/677	500/8 ppm	6250	6172	32	16,832	6216	6140	60	17,332
Nalco GWP 827/677	300/8 ppm	7376	7296	36	15,998	7376	7308	36	16,332
Nalco 8103	3000 ppm	6816	6680	88	18,665	6788	6500	224	17,665
Nalco 8103	1000 ppm	7280	7172	64	21,331	7260	7072	152	21,431
Nalco 8103/677	500/8 ppm	7316	7232	44	19,265	7244	7124	80	19,398
Nalco 107/677	500/8 ppm	5436	5408	32	15,832	5420	5412	12	15,832

TABLE E-2

PHYSICAL DATA FROM NALCO 107 POLYMER TRIAL (10/5/77 & 10/6/77)

Sample	pH	Suspended Solids (ppm)	Total Solids (ppm)	Color
(11:30 p.m. 10/5/77)				
Caustic Extract	10.0	64	6810	17,332
After Vac-20	8.6	2768	8066	5.999
After Broughton	8.5	1124	7622	6,632
Permeate	8.6	4	5470	1,733
Concentrate	8.5	1068	2686	66,332
(3:30 a.m. 10/6/77)				
Caustic Extract	11.0	96	6734	17,498
After Vac-20	9.1	2828	7710	5,999
After Broughton	8.9	1160	7176	6,499
Permeate	9.0	8	5298	1,667
Concentrate	9.0	1024	7186	6,366
(10:00 a.m. 10/6/77)				
Caustic Extract	---	116	6938	19,998
After Vac-20	7.5	2164	8090	3,000
After Broughton	8.8	1000	7370	6,499
Permeate	9.5	36	6100	4,700
Concentrate	8.9	972	7322	6,666
(3:00 p.m. 10/6/77)				
Caustic Extract	10.5	---	8142	17,332
After Vac-20	9.2	916	9464	8,332
After Broughton	10.4	1204	8700	8,333
Permeate	8.4	---	6206	3,066
Concentrate	8.2	928	8562	7,833

TABLE E-3

FLUX DECLINE VERSUS TIME FOR NALCO 107 TRIAL

Elapsed Running Time (Hrs.)	Flux (gfd)	Percent Flux Loss
0	38.5	0
1	25.2	34.5
2.7	14.7	61.8
3.3	11.4	70.3
4.1	8.6	77.7
5.1	7.6	80.3
6.1	6.2	83.9
7.1	5.2	86.5
8.1	4.8	87.5
9.1	3.8	90.1

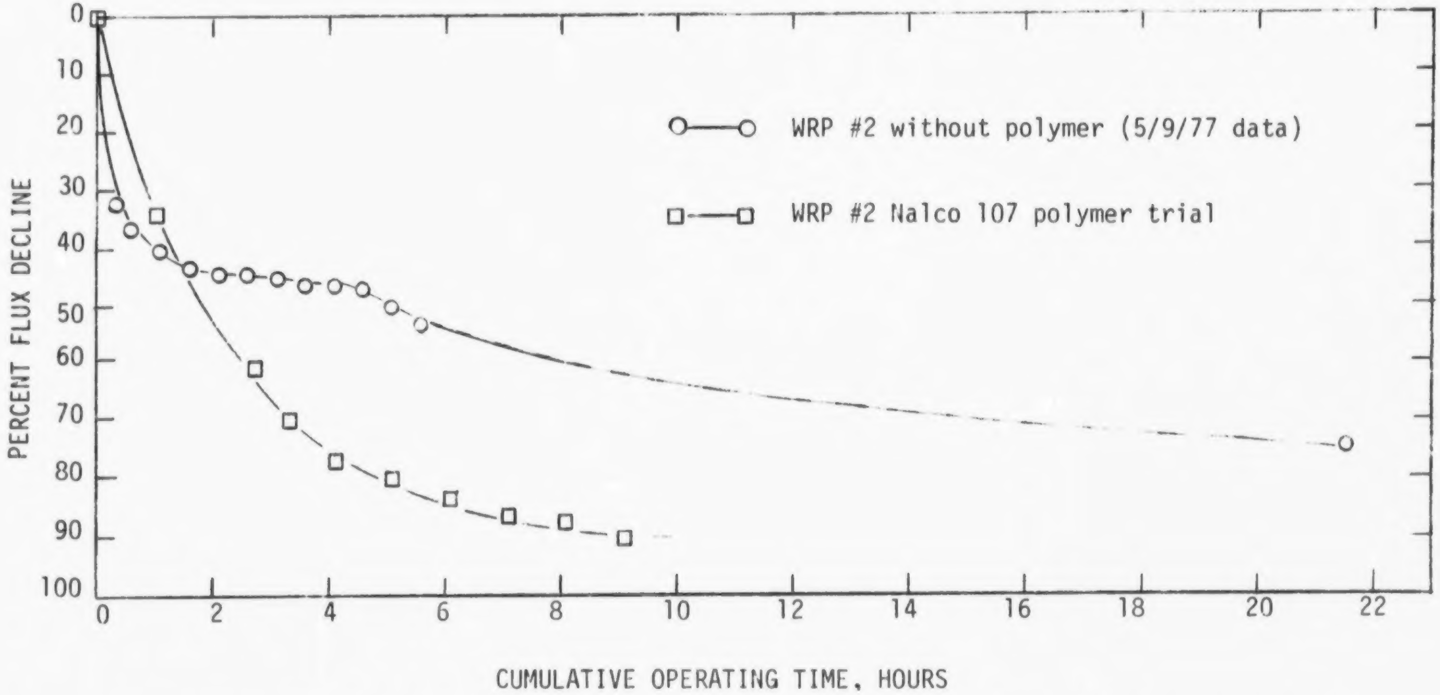


Figure E2. WRP #2 module flux decline with and without Nalco 107 polymer pretreatment.

Table E4 contains additional data from the trial. After 20 hours of running, five hours after the Nalco 7763 flocculant addition was started, the membrane module was given a one-hour cleaning with ultraclean solution. The hoped for flux recovery was not obtained with this quick cleaning. The flux of 0.29 m^3/m^2-day (7.1 gfd) rapidly decreased back to the 0.08 m^3/m^2-day (1.9 gfd) value of before cleaning.

51 MM DIAMETER DEPTH FILTER PRETREATMENT STUDIES

A 51 mm (2 in) diameter depth filter was operated at the Canton Mill to assess the performance of various filter media. Representative data from runs with beds of garnet sand, anthracite coal and silica sand, filter AG and granular PVC are given in Tables E5 through E8. No media was significantly superior to the filter AG (used in the Kisco filters) in terms of suspended solids removal.

All media tested in the 51 mm diameter column exhibited substantial head loss over relatively short periods of time (1 to 6 hours). This occurred even though the caustic extraction filtrate feed stream was pretreated by a hydrasieve for fiber removal.

Based on these tests no change in the Kisco filter media was made.

TABLE E-4

OPERATING DATA FROM NALCO 107 TRIAL

	Time	Elapsed Running Time (Hrs.)	Flux (gfd)	Pressure Drop Across Module (psig)	Total Flow Rate (gpm)
10/5	11:25 am	0	38.5	32	12
	12:25 pm	1	25.2	41	15
	2:05	2.7	14.7	21	10
	2:45	3.3	11.4	15	8
	3:30	4.1	8.6	32	14
	4:30	5.1	7.6	32	14
	5:30	6.1	6.2	34	14
	6:30	7.1	5.2	34	14
	7:30	8.1	4.8	34	14
	8:30	9.1	3.8	34	14
	9:30	10.1	3.8	34	14
	10:25	11.0	3.3	35	14
	11:30	12.1	2.9	34	14
10/6	12:30 am	13.1	2.7	34	14
	1:30	14.1	2.5	34	14
	2:30	15.1	2.4	34	14
	3:30	16.1	1.9	34	14
	4:30	17.1	1.9	34	14
	5:30	18.1	2.1	35	14
	6:30	19.1	1.9	35	14
	7:30	20.1	1.9	35	14
	8:30	System shut down for cleaning with ulatraclean solution			
	10:00	0	7.1	40	13
	11:00	1	4.8	42	15
	12:00 pm	2	3.8	42	15
	12:55	2.9	3.0	42	15
	2:00	4	2.9	40	14
	3:00	5	2.3	40	14
	4:00	6	2.1	40	14
	5:00	7	2.1	40	14
	6:00	8	1.9	40	14
	6:45	8.75	1.9	39	14

TABLE E5. REPRESENTATIVE OPERATING DATA FOR 51 MM DIAMETER DEPTH FILTER WITH 0.75 M BED DEPTH OF GARNET SAND*

Date	Cumulative operating time, hrs	Feed suspended solids, mg/l	Filtrate suspended solids, mg/l	Removal efficiency, %
5/3	0.7	48	26	45.8
	3.5	48	18	62.5
	4.5	44	20	54.5
	5.5	40	18	55.0
5/4	0.5	64	18	71.9
	2.5	40	14	65.0
	4.5	30	8	73.3
5/5	1.5	46	22	52.2
	2.5	40	18	55.0
	4.5	38	18	52.6
5/6	1.5	40	22	45.0
	2.5	38	22	42.1
	3.5	38	22	42.1
	6.5	34	22	35.3
5/9	1.5	50	28	44.0
	2.5	48	30	37.5
	4.5	34	26	35.3
	5.5	24	80	--
5/10	1.0	54	52	4.7
	2.5	58	30	31.0

---

* Feed stream is caustic extraction filtrate pretreated by a hydrasieve.

TABLE E6. REPRESENTATIVE OPERATING DATA FOR 51 MM DIAMETER DEEP BED FILTER WITH 0.51M ANTHRACITE COAL PACKED OVER 0.25M SILICA SAND*

Date	Cumulative operating time, hrs	Feed suspended solids, mg/l	Filtrate suspended solids, mg/l	Removal efficiency, %
2/16	0.33	48	20	58.3
2/18	0.08	76	18	76.3
	0.75	36	14	61.1
2/22	0.33	72	36	50.0
	1.08	78	42	46.2
	2.13	84	36	57.1
	3.08	56	28	50.0
	4.08	42	12	71.4
	5.08	64	18	71.9
	5.88	116	52	55.2
	6.88	104	48	53.8
	7.88	56	16	71.4
	8.88	84	32	61.9
	9.88	84	38	54.8
2/24	0.5	82	38	53.7
	1.5	60	30	50.0
	2.5	52	26	50.0
	3.5	70	66	5.7
	4.5	66	52	21.2
2/28	.33	106	34	67.9
	1.33	98	40	59.2
	2.33	100	30	70.0
	3.33	106	62	41.5
	4.33	98	52	46.9
	5.33	100	48	52.0
	5.83	80	44	45.0
3/2	.67	52	38	26.9
	2.67	58	28	51.7
	2.75	60	30	50.0
	3.67	44	14	68.2
	4.67	50	28	44.0

* Feed stream is caustic extraction filtrate pretreated by a hydrasieve.

TABLE E7. REPRESENTATIVE OPERATING DATA FOR 51 MM DIAMETER DEEP BED FILTER WITH 0.75 M BED DEPTH OF FILTER AG*

Date	Cumulative operating time, hrs	Feed suspended solids, mg/l	Filtrate suspended solids, mg/l	Removal efficiency, %
3/7	2.33	46	22	52.2
	3.33	64	22	65.6
	4.33	86	44	48.8
3/8	.25	66	18	72.7
	1.25	64	14	78.1
	2.25	72	40	44.4
	3.25	80	40	50.0
3/10	.35	90	42	53.3
	1.42	82	46	43.9
	2.42	84	50	40.5
	3.42	84	48	42.9
3/14	.58	40	32	20.0
	1.58	42	28	33.3
	2.58	40	12	70.0
	3.58	48	16	66.7
	4.58	66	16	75.8
	5.58	42	20	52.4
3/16	.75	68	36	47.1
	1.75	64	16	75.0
	2.75	52	4	92.3
	3.75	36	4	88.9
	4.75	44	4	90.9
3/24	.58	80	44	45.0
	1.56	76	30	60.5
	2.58	74	32	57.8
	3.58	64	16	75.0
	4.58	42	12	71.4
	5.58	64	20	68.8
	6.08	64	22	65.6
	7.03	180	36	80.0
	7.53	70	26	62.9
	9.03	62	20	67.7
	10.03	54	22	59.3
	12.03	76	26	65.8

* Feed stream is caustic extraction filtrate pretreated by a hydrasieve.

TABLE E8. REPRESENTATIVE OPERATING DATA FOR 51 MM DIAMETER DEPTH FILTER WITH 0.75 M BED DEPTH OF GRANULAR PVC*

Date	Cumulative operating time, hrs	Feed suspended solids, mg/l	Filtrate suspended solids, mg/l	Removal efficiency, %
4/1	1.11	74	24	67.6
	1.67	82	36	56.1
	2.17	72	40	44.4
	2.67	70	64	14.3
	3.17	68	42	38.2
	3.67	102	50	51.0
	4.17	80	32	60.0
	4.67	64	20	68.8
4/4	5.67	62	24	61.3
	6.67	74	36	51.4
	7.67	60	40	33.3
	8.67	52	28	46.2
	9.67	60	24	60.0
	11.17	96	40	58.3
	12.17	118	46	61.0
	13.17	96	64	33.3
	14.67	80	40	50.0
	15.67	40	8	80.0
4/6	17.58	62	4	93.5
	18.58	56	2	96.4
	20.08	52	12	76.9
	21.08	48	2	95.8
	22.08	64	8	87.5
	23.91	76	16	78.9
	25.41	76	18	76.3
	26.41	72	20	72.2

* Feed stream is caustic extraction filtrate pretreated by a hydrasieve.

LETTER FROM J.A. NOWAK, NALCO

DATE: JULY 14, 1977

SUBJECT: PLUGGED CHAMPION PAPERS

The plugging of Champion Paper ultra-filtration membranes can be attributed to particles of kaolinite and titanium dioxide which are coated and agglomerated with starch. The agglomerated particles also contain a carboxylic acid salt which may stabilize the colloidal nature of the foulant.

Scanning electron micrographs of a new membrane and a plugged membrane are shown in Figures E3 and E4 respectively. The micrograph of the new membrane shows that the membrane pore size is very small (less than 0.1 microns) and that the surface of the plugged membrane is completely covered with the "slime" foulant. Figures E5 and E6 are microprobe results which indicate the chemical composition of the "slime".

Transmission electron micrographs of the "slime" sample are shown in Figures E7, E8 and E9. The large plate-like particles in Figure E7 are identified as kaolinite clay, while the small dense particles are titanuim dioxide. The micrograph also indicates the presence of a coating surrounding the particles and holding them together into larger chumps. Figures E8 and E9 further illustrate the degree to which the particles are coated and agglomerated.

X-ray diffraction and X-ray fluorescence analysis of the dried slime revealed it to contain kaolinite ($Al_2Si_2O_5(OH)_4$) and anatase (TiO_2). Infrared spectroscopy indicated that the major organic constituent was starch. The presence of starch was confirmed by chemical spot tests. A carboxylic acid salt was also found to be present at low concentrations. The composition of the slime is best represented as follows:

Volatiles at 105°C - 88.0%
Kaolinite Clay - 5.6%
Starch - 4.0%
Titanium dioxide - 1.4%
Carboxylic acid salt - <1.0%

The combination of the small pore size of the membrane, together with the starch coated and colloidal nature of the particles, would seriously impair the performance of the ultra-filtration membranes.

Please contact me if you require additional information or work.

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Figure E3. New membrane 5000X.

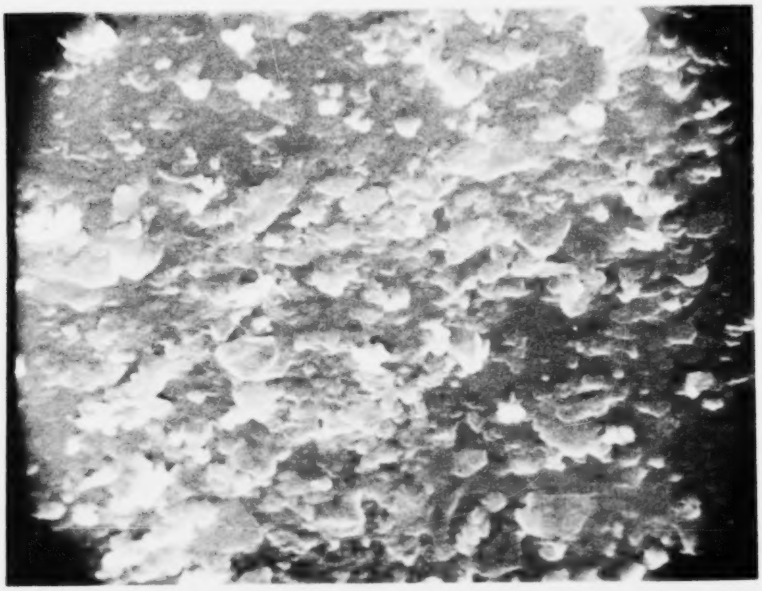

Figure E4. Fouled membrane 5000X.

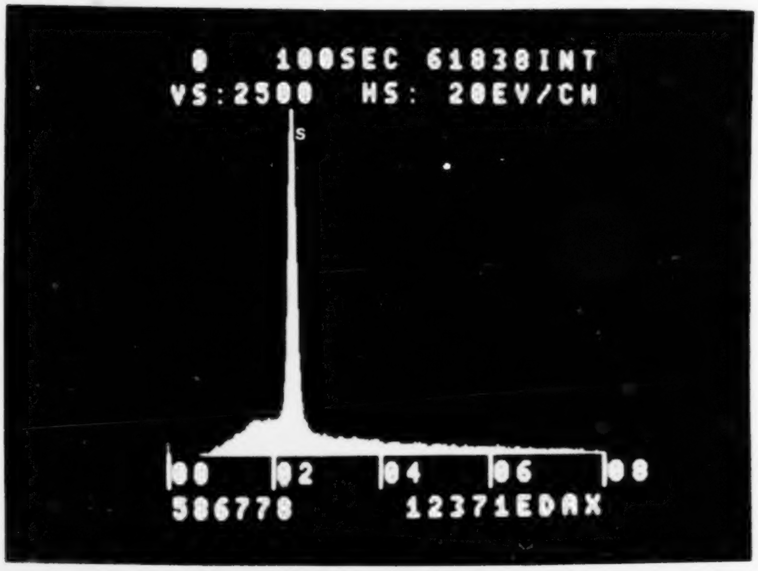

Figure E5. Elemental analysis of new membrane.

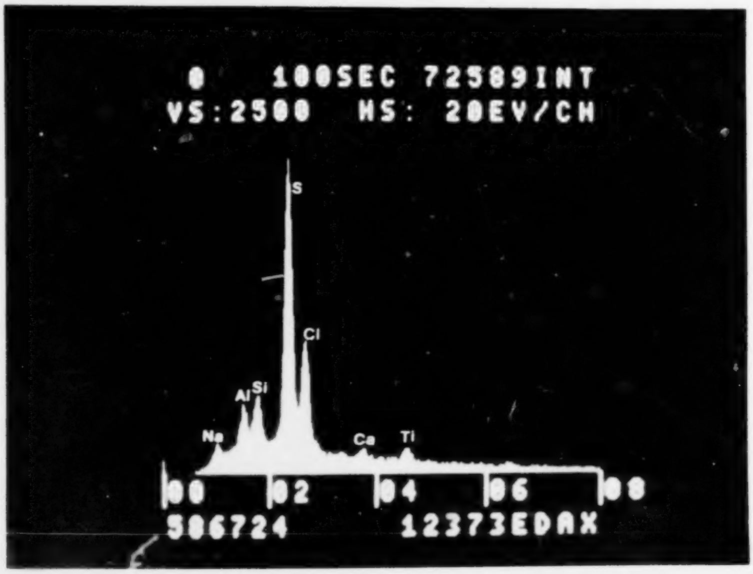

Figure E6. Elemental analysis of fouled membrane.

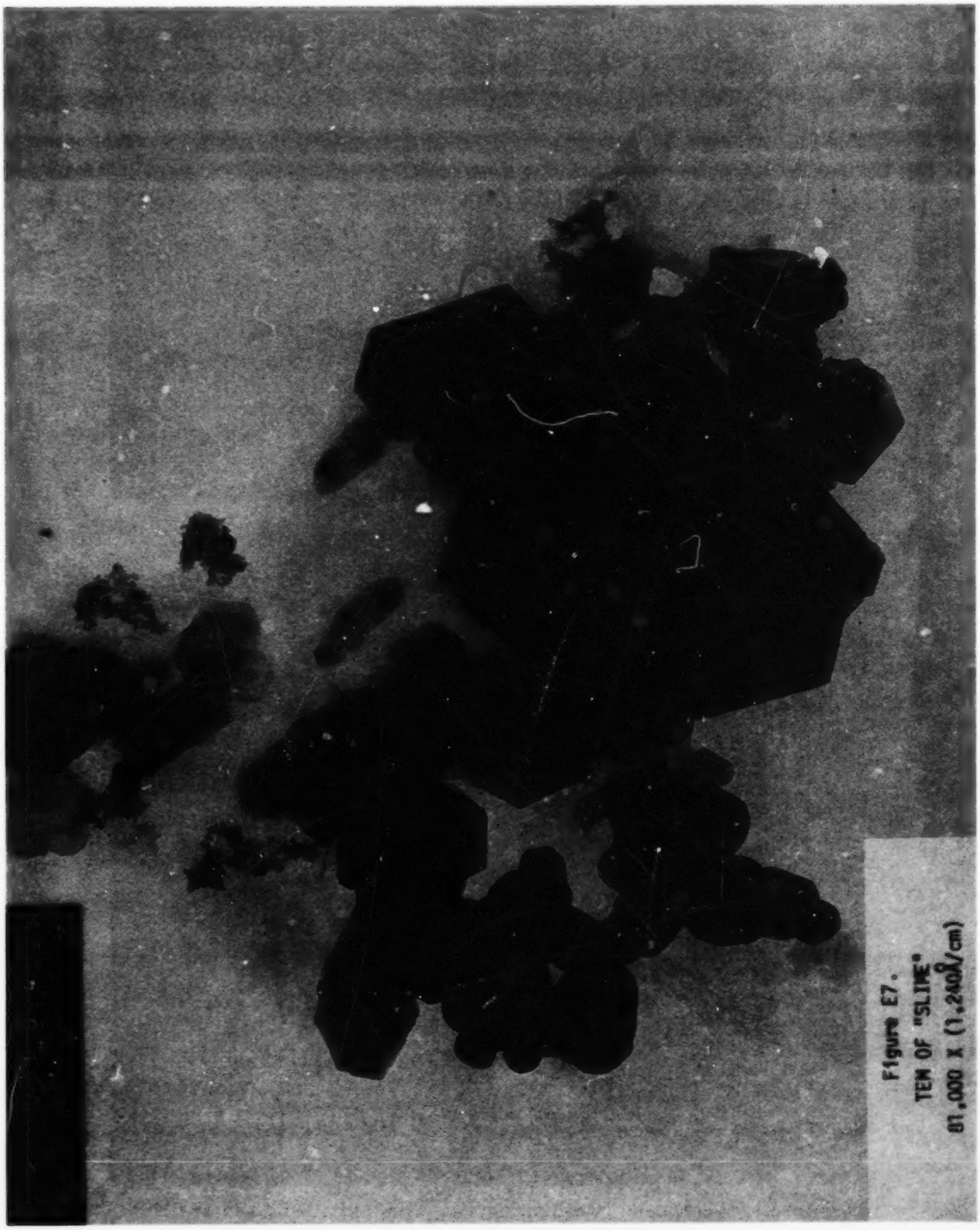

Figure E7.
TEM OF "SLIME"
81,000 X (1,240Å/cm)

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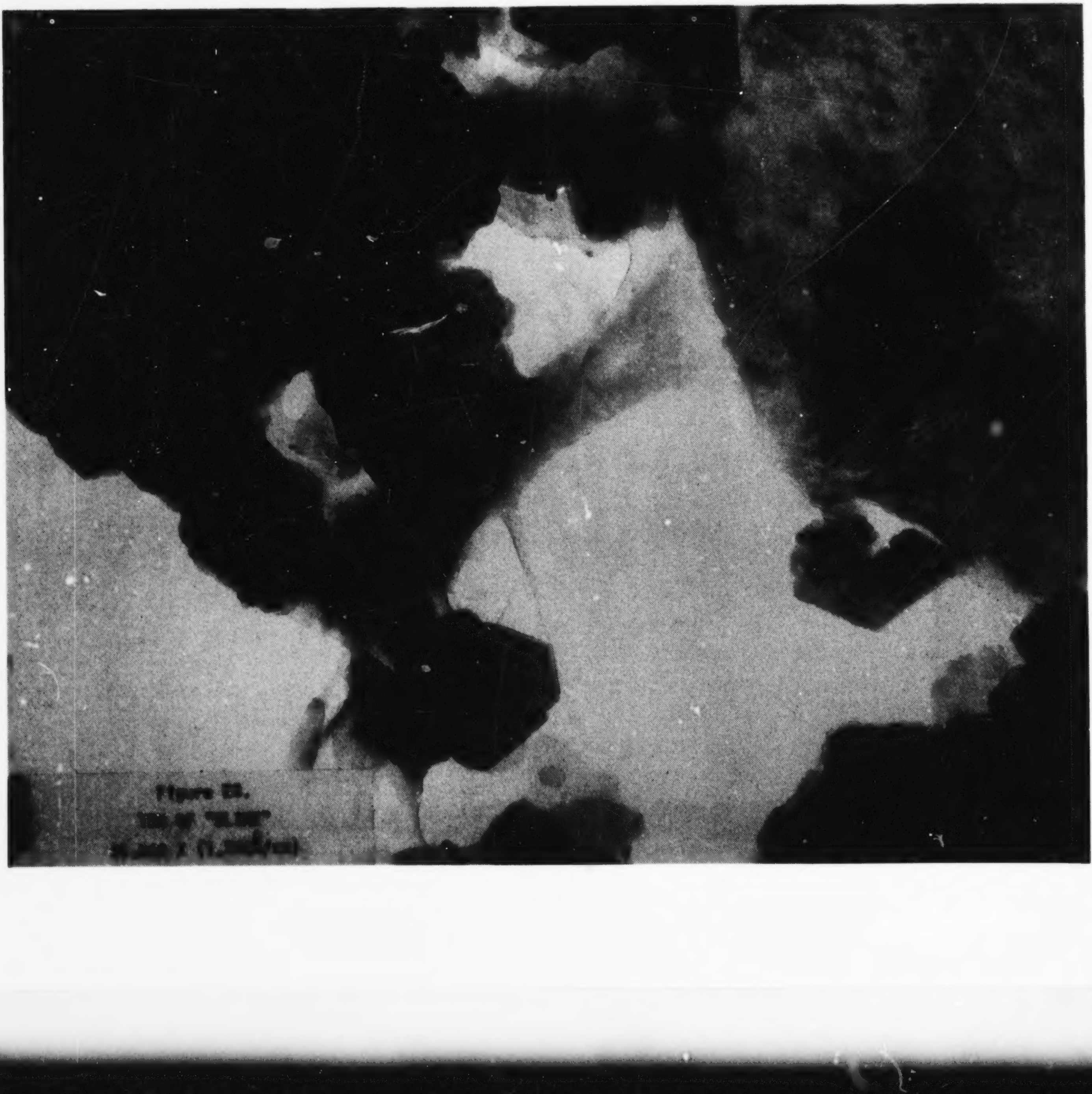


Figure 55.
TEM OF "[illegible]"
30,000 X [illegible]

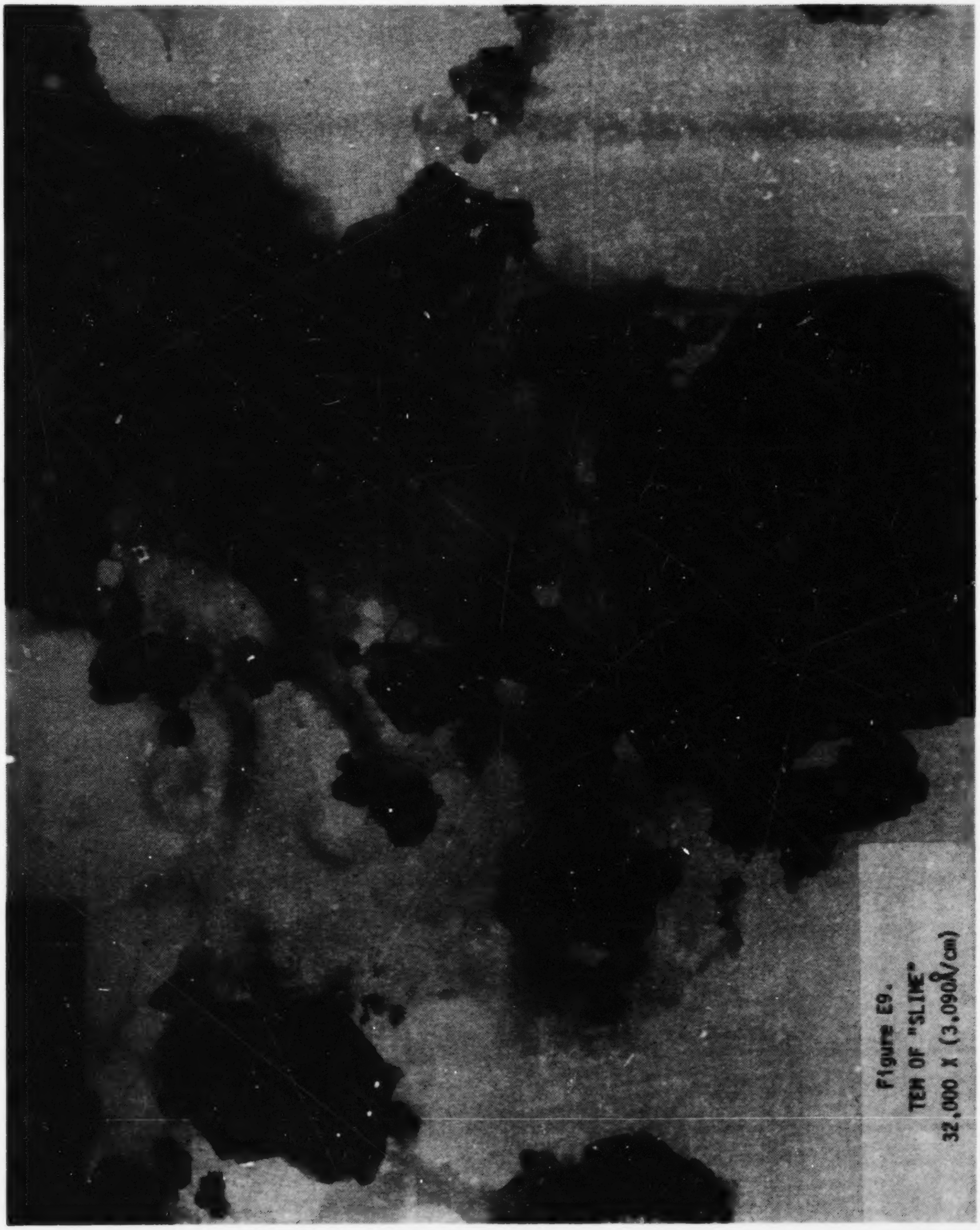


Figure E9.
TEM OF "SLIME"
32,000 X (3,090Å/cm)

APPENDIX F. 3-STAGE PILOT SYSTEM COLOR REJECTION DATA

TABLE F1. FEED, RECYCLE, REJECT AND PERMEATE COLOR CONCENTRATIONS DURING 3-STAGE PILOT SYSTEM TESTS (COLOR UNITS)

Cumulative operating time (hours)	Feed (after broughton)	Stage 1 Recycle	Stage 1 Reject	Stage 1 Permeate	Stage 2 Recycle	Stage 2 Reject	Stage 2 Permeate	Stage 3 Recycle	Stage 3 Reject	Stage 3 Permeate
2	23,331	27,331	--	3,966	32,997	--	4,666	59,327	70,993	6,299
5.5	--	25,666	--	4,900	26,997	--	4,433	53,328	59,661	4,766
23	18,665	19,998	--	3,100	21,998	--	1,633	25,997	27,330	1,333
30	17,332	22,998	--	2,633	27,331	--	3,400	173,983	189,981	9,699
48	20,731	25,664	--	6,166	29,660	--	4,300	74,993	74,973	4,666
49	17,299	21,331	--	3,666	31,630	--	3,400	59,661	40,663	4,666
69	18,665	24,664	--	5,533	36,663	--	5,333	100,657	106,323	9,732
75	17,332	21,998	--	5,133	30,997	--	5,000	74,659	79,654	8,732
83	14,999	21,331	--	6,399	47,329	44,996	5,666	--	--	--
90	17,332	21,331	--	5,999	33,663	39,663	20,998	--	--	--
97	17,998	23,166	--	5,000	49,995	52,995	6,333	--	--	--
120	16,665	18,665	--	5,000	30,997	30,997	3,666	--	--	--
139	18,332	19,765	--	4,500	28,331	29,997	2,400	--	--	--
141	17,998	20,831	--	5,299	39,996	43,996	3,666	--	--	--
142	15,065	17,965	--	6,066	53,328	56,661	5,666	--	--	--
148	18,665	22,664	--	7,666	89,658	91,658	7,966	--	--	--
167*	3,400	4,300	--	1,033	12,332	12,665	467	--	--	--
175*	3,166	3,666	--	1,966	15,998	16,665	833	--	--	--
177	14,979	18,065	--	7,099	64,994	69,660	5,633	--	--	--
197	19,998	25,331	--	13,665	84,325	89,991	8,632	--	--	--
198	14,832	20,998	--	3,500	34,996	--	5,000	133,320	166,317	25,331
206	--	33,997	--	4,500	53,328	--	8,366	227,311	256,641	30,664
217	16,332	24,998	--	3,033	54,995	--	7,266	206,646	256,641	22,664
219	13,499	19,665	22,331	2,666	41,663	44,996	4,600	154,985	158,318	11,832
232	25,331	33,330	39,996	8,333	108,323	114,989	13,332	--	996,567	59,994
235	24,331	26,664	27,331	9,066	59,327	64,660	8,166	219,978	222,978	--
239	22,331	28,997	29,664	5,966	64,994	70,160	8,832	266,640	283,305	20,665
246	14,965	18,498	16,665	4,366	48,329	49,995	5,099	219,978	236,664	14,999
249	20,665	26,664	29,997	7,433	59,994	66,993	8,166	219,978	226,644	16,665
255	20,998	27,997	28,664	7,499	96,657	108,323	11,832	109,989	139,986	--
275	25,997	32,997	36,663	9,499	61,661	64,994	8,666	198,314	216,645	12,832
282	16,336	20,498	20,998	4,500	39,996	44,996	6,633	141,653	143,319	10,999
300	21,998	25,331	25,664	8,166	36,663	42,662	12,299	193,314	209,979	11,166
320	16,332	16,998	17,665	10,832	40,329	42,996	7,666	146,319	161,651	5,066
325	26,997	39,663	42,652	9,666	83,325	84,325	15,498	316,635	329,967	39,663
335	17,932	25,997	28,331	5,966	59,661	60,661	10,999	273,306	293,304	30,664

* Processing pine decker effluent

TABLE F2. WRP SPIRAL-WOUND MODULE COLOR REJECTION DURING 3 STAGE PILOT PLANT TESTS

	Stage 1		Stage 2		Stage 3	
Cumulative operating time (hours)	% rejection feed basis	% rejection concentrate basis	% rejection feed basis	% rejection concentrate basis	% rejection feed basis	% rejection concentrate basis
2	83.0	85.5	80.0	85.9	73.0	89.4
5.5	--	80.9	--	83.6	--	91.0
23	83.4	84.5	91.3	92.5	92.9	94.9
30	84.8	88.6	80.4	87.6	44.0	94.4
48	70.3	76	79.3	85.5	77.5	93.8
49	78.8	83.8	80.3	89.3	73.0	92.2
69	70.4	76.0	71.4	85.5	47.9	90.3
75	70.4	76.7	71.1	83.9	49.6	88.3
83	57.3	70.0	62.2	88.0	--	--
90	65.4	71.9	0	37.6	--	--
97	72.2	78.4	64.8	87.3	--	--
120	70.0	73.2	78.0	88.2	--	--
139	75.5	77.2	86.9	83.7	--	--
141	71.1	74.6	80.0	91.6	--	--
142	59.7	66.2	62.4	89.4	--	--
148	58.9	66.2	57.3	91.1	--	--
167*	69.6	76.0	86.3	96.2	--	--
175*	37.9	46.4	73.7	94.8	--	--
177	52.6	61.3	62.4	91.3	--	--
197	31.7	46.1	56.8	89.8	--	--
198	76.4	83.3	66.3	85.7	0	81.0
206	--	87.9	--	84.3	--	86.5
217	81.4	87.9	55.5	86.8	0	89.0
219	80.3	86.4	65.9	89.0	12.3	92.4
232	67.1	72.0	47.4	87.7	0	--
235	62.7	66.0	66.4	86.2	--	--
239	73.3	79.4	60.4	86.4	7.5	92.2
246	70.8	76.4	65.9	89.4	0	93.2
249	64.0	72.1	60.4	86.4	19.4	92.4

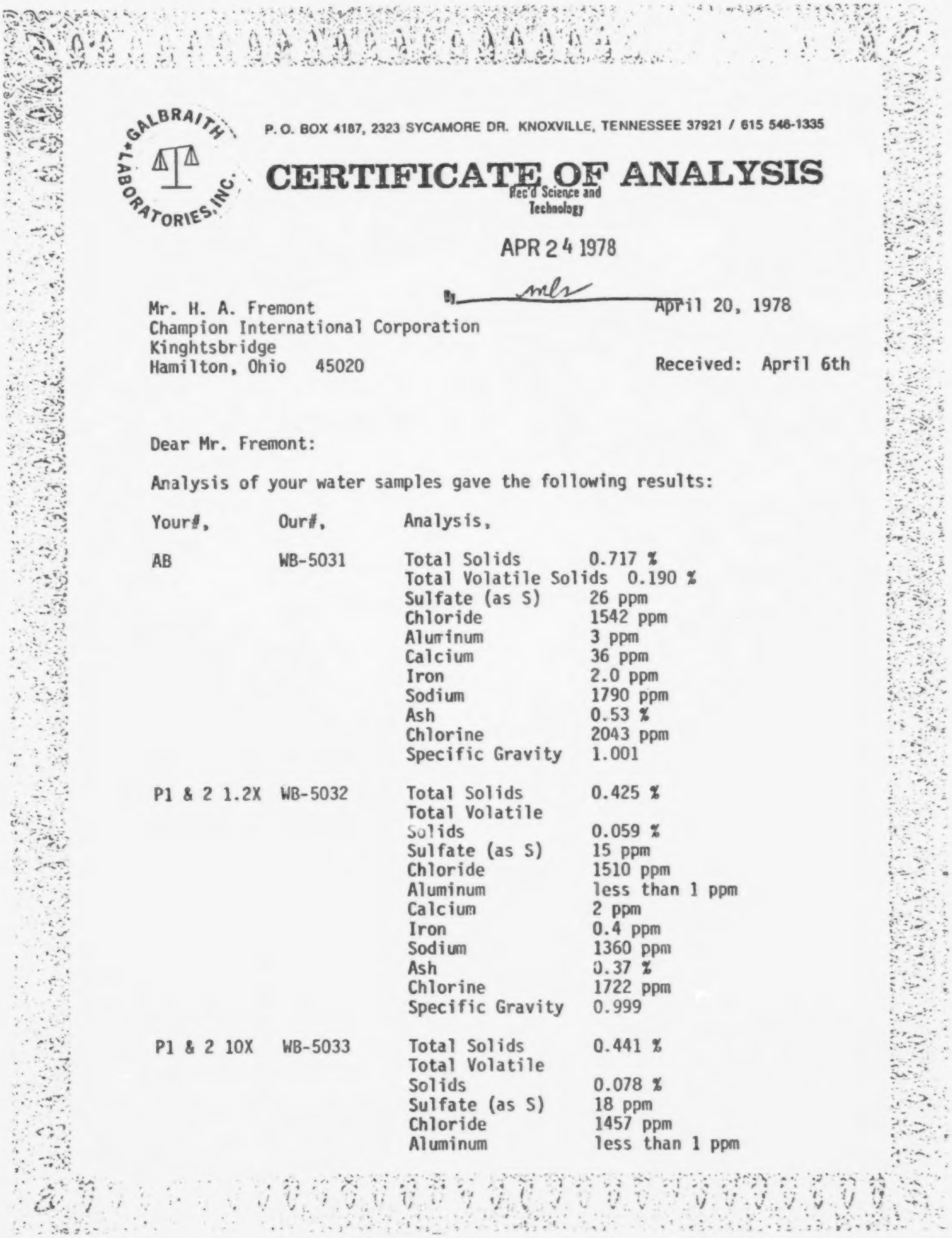
(continued)

TABLE F2 (continued)

	Stage 1		Stage 2		Stage 3	
Cumulative operating time (hours)	% rejection feed basis	% rejection concentrate basis	% rejection feed basis	% rejection concentrate basis	% rejection feed basis	% rejection concentrate basis
255	64.3	73.2	43.7	87.8	--	--
275	63.5	71.2	66.7	85.9	50.7	93.5
282	72.4	78.0	59.4	83.4	32.7	92.2
300	62.9	67.8	44.1	66.5	49.2	94.2
320	33.7	36.3	53.1	81.0	69.0	96.5
325	64.2	75.6	42.6	81.4	0	87.5
335	66.7	77.1	38.7	81.6	0	88.8

* Processing pine decker effluent

APPENDIX G. ANALYTICAL DATA FROM 25.4 MM DIAMETER TUBULAR ASSEMBLY EXPERIMENTS



GALBRAITH LABORATORIES, INC.
P. O. BOX 4187, 2323 SYCAMORE DR. KNOXVILLE, TENNESSEE 37921 / 615 546-1335

CERTIFICATE OF ANALYSIS

Rec'd Science and Technology
APR 24 1978
By mls

Mr. H. A. Fremont
Champion International Corporation
Kinghtsbridge
Hamilton, Ohio 45020

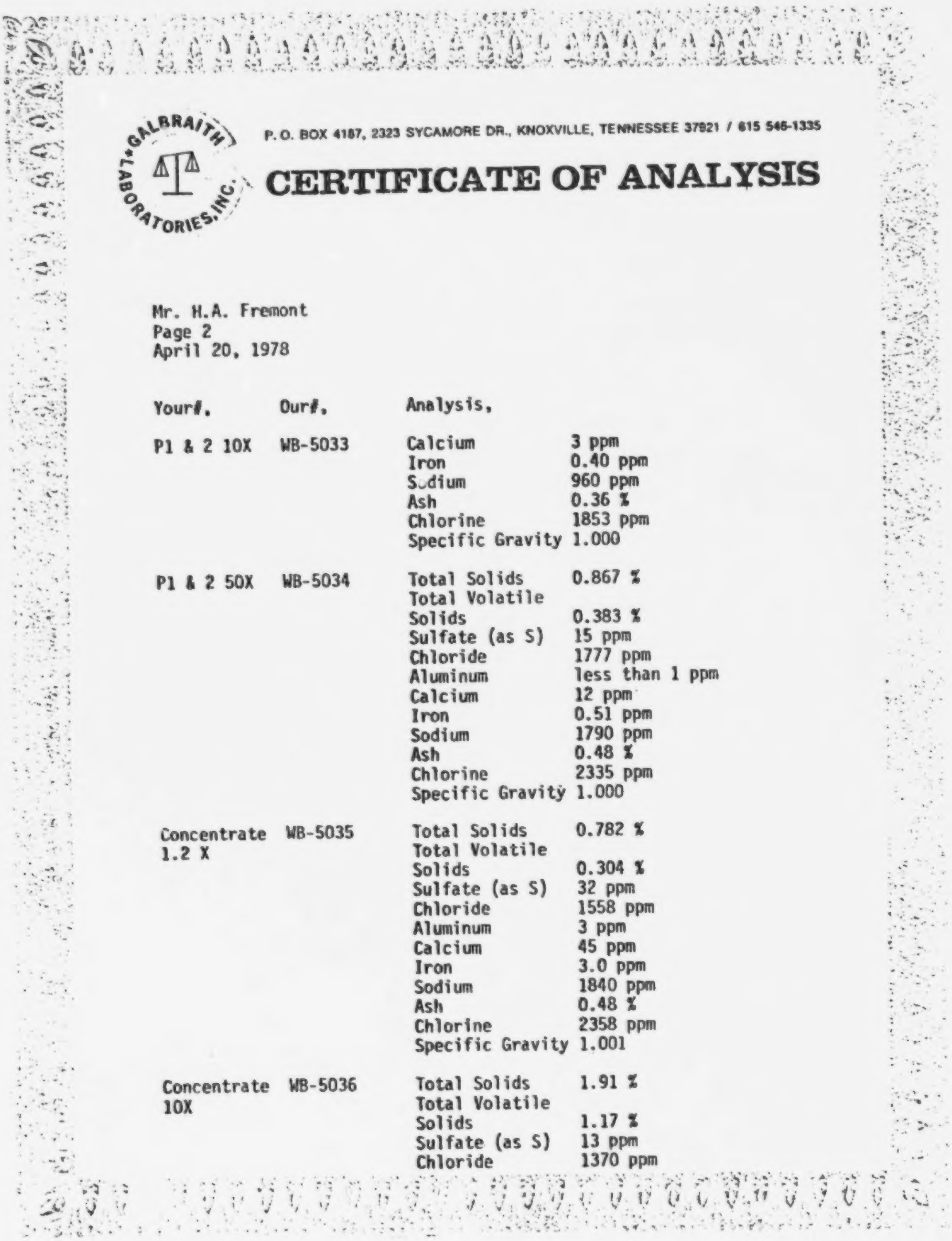
April 20, 1978

Received: April 6th

Dear Mr. Fremont:

Analysis of your water samples gave the following results:

Your#,	Our#,	Analysis,	
AB	WB-5031	Total Solids	0.717 %
		Total Volatile Solids	0.190 %
		Sulfate (as S)	26 ppm
		Chloride	1542 ppm
		Aluminum	3 ppm
		Calcium	36 ppm
		Iron	2.0 ppm
		Sodium	1790 ppm
		Ash	0.53 %
		Chlorine	2043 ppm
		Specific Gravity	1.001
P1 & 2 1.2X	WB-5032	Total Solids	0.425 %
		Total Volatile Solids	0.059 %
		Sulfate (as S)	15 ppm
		Chloride	1510 ppm
		Aluminum	less than 1 ppm
		Calcium	2 ppm
		Iron	0.4 ppm
		Sodium	1360 ppm
		Ash	0.37 %
		Chlorine	1722 ppm
		Specific Gravity	0.999
P1 & 2 10X	WB-5033	Total Solids	0.441 %
		Total Volatile Solids	0.078 %
		Sulfate (as S)	18 ppm
		Chloride	1457 ppm
		Aluminum	less than 1 ppm



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Your#.	Our#.	Analysis,	
P1 & 2 10X	WB-5033	Calcium	3 ppm
		Iron	0.40 ppm
		Sodium	960 ppm
		Ash	0.36 %
		Chlorine	1853 ppm
		Specific Gravity	1.000
P1 & 2 50X	WB-5034	Total Solids	0.867 %
		Total Volatile Solids	0.383 %
		Sulfate (as S)	15 ppm
		Chloride	1777 ppm
		Aluminum	less than 1 ppm
		Calcium	12 ppm
		Iron	0.51 ppm
		Sodium	1790 ppm
		Ash	0.48 %
		Chlorine	2335 ppm
		Specific Gravity	1.000
Concentrate 1.2 X	WB-5035	Total Solids	0.782 %
		Total Volatile Solids	0.304 %
		Sulfate (as S)	32 ppm
		Chloride	1558 ppm
		Aluminum	3 ppm
		Calcium	45 ppm
		Iron	3.0 ppm
		Sodium	1840 ppm
		Ash	0.48 %
		Chlorine	2358 ppm
		Specific Gravity	1.001
Concentrate 10X	WB-5036	Total Solids	1.91 %
		Total Volatile Solids	1.17 %
		Sulfate (as S)	13 ppm
		Chloride	1370 ppm

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CERTIFICATE OF ANALYSIS

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Your#,	Our#,	Analysis,	
Concentrate 10 X	WB-5036	Aluminum	16 ppm
		Calcium	241 ppm
		Iron	18 ppm
		Sodium	2650 ppm
		Ash	0.74 %
		Chlorine	3487 ppm
		Specific Gravity	1.005
Concentrate 50X	WB-5037	Total Solids	10.28 %
		Total Volatile Solids	8.22 %
		Sulfate (as S)	4.6 ppm
		Chloride	1015 ppm
		Aluminum	152 ppm
		Calcium	1180 ppm
		Iron	92 ppm
		Sodium	6510 ppm
		Ash	2.07 %
		Chlorine	8766 ppm
		Specific Gravity	1.044

Sincerely yours,

GALBRAITH LABORATORIES,INC.

Gail R. Hutchens
Gail R. Hutchens
Exec Vice-President

GRHyk